

**A Draft Report[©] for the
Pilot Demonstration of**

***Controlled In-Situ Chemical Oxidation
Technology***

Cool-Ox[™]
at

**Ashland/Xcel Energy Lakefront Site
d.b.a Xcel Energy, a subsidiary of Xcel Energy ("NPSW")
Ashland, WI**

June 1, 2007

**DTI Project # 1162-R2
Joint Project**

Prepared for

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June 1, 2007

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Re: A Draft Report for the Application of Cool-Ox™ Controlled In-Situ Chemical Oxidation Technology at Ashland/Xcel Energy Lakefront Site, d.b.a. Xcel Energy, a subsidiary of Xcel Energy (“NSPW”), Ashland, WI, DTI/DCI Joint Project # 1162-R2

Dear Mr. Winslow:

Attached please find the DCI/DTI (TEAM) Joint Project Draft Report outlining the activities and findings of the *Cool-Ox™* in-situ chemical oxidation (ISCO) Pilot Demonstration conducted at subject site under the direction of the USEPA SITE Demonstration Program. The Report has been compiled based upon information collected from all participants including Field Supervisors and Technicians, the EPA technical team, Consultants, WDNR personnel, Coleman Engineering and others.

Because the USEPA Site Team had not previously conducted a demonstration project involving an on-going dynamic technology, they were concerned that as many aspects of the study as possible, be examined. This included information developed from the January and April (2007) sampling events subsequent to injection work. Therefore, time was necessary to evaluate and assimilate those findings into this document.

(Frank please add your comments)

Acknowledgements:

The DCI/DTI TEAM (TEAM) is most grateful to personnel from Newfields, Coleman, Tetra-Tech, the USEPA and the WDNR for their contribution of information and exceptional cooperation during the execution of this project. Although many obstacles were encountered requiring the modification of the Scope of Work (SOW) as well as the Quality Assurance Project Plan (QAPP), the TEAM enjoyed a genuine sense of the “*spirit of curiosity and cooperation*” and the definite “*desire to understand the nuances of this new Technology*” from all those involved. A special thank you is reserved for the goodwill and support of the local NSPW staff and employees at the site. Their assistance and cooperation was key to the successful completion of the work.

Forward:

In his poem “*To a Mouse*”, the eighteenth-century Scottish poet, Robert Burns penned the cryptic reference to the brevity of the “*best laid schemes o’ mice an’ men.*” This phrase can certainly be applied to the SOW and QAPP designed to direct the execution of this pilot demonstration. From the beginning it became apparent that work activities based upon historical records and the old Sanborn Maps must be modified to speak to current site conditions. Injection activities in both treatment zones (MW-15 and MW-13), located structures that had been described in the old records as having been removed. The presence of these artifacts and the associated unexpected masses of contaminants dramatically changed the anticipated scope of the Technology application as well as the monitoring procedures of the QAPP. These points were discussed in the teleconferences and on-going amendments, based upon frequent new information derived from the dynamic nature of the Technology, were incorporated into the SOW. In the end, these open discussions resulted in a communication conduit that spoke to the concerns of all parties.

Three discoveries were made during the site work that dramatically changed the course of the demonstration project. These were that:

1. the location of the Filled Ravine was actually located to the west of the MW-15 demonstration area, placing it beyond the influence of the Technology,
2. the MW-15 monitoring well was actually located inside of a gas holder that the historical record had reported to have been removed, (this actually turned out to be a critical benefit to the project), and;
3. the Copper Falls Aquifer contained a much higher concentration of rock and cobbles than was anticipated which greatly increased the difficulty in penetrating to design depth.

The affect of this newly developed information, based upon observations made during the application activities in both pilot areas as well as the January and April sampling events at the MW-15 monitoring well nest, were dramatic. The information collected under-pinned the observations made at previous MGP applications and provided the validation of the Technology as a useful remedial tool in this industry.

As such, interpretation of the sampling and analytical data collected during the January and April sampling events is critical to the technical understanding of the effect of the Technology on the contaminants as well as the observations made for the entire pilot demonstration. Because of the complex turn of events during the course of the project and the need to attempt to clearly understand the associated ramifications, additional time was necessary to interview all participants and assimilate as much information as possible. TEAM members experienced in numerous applications of the *Cool-Ox*TM Technology, have had several communications with the USEPA, WDNR, Tetra-Tech, Newfields and Coleman project personnel in effort to provided practical insight into the project and mutually develop an understanding of the results. This report will attempt to present the findings as they were encountered and the activities necessary to bring the project to a useful conclusion.

Background:

Initially, the proposed *Cool-Ox*TM Technology pilot demonstration project conducted at NSPW, called for treating only the area around the **MW-15** monitoring well nest. The purpose of the project was to demonstrate the ability of the Process to validate itself in three areas. These were, its capability to:

1. locate contaminant sources,
2. reduce the concentrations of contaminants, and;
3. stimulate and accelerate the proliferation of intrinsic microbial degraders.

In short, the project was designed to validate the Technology in areas where it had already been proven successful. However, this program was not congruent with the interests of WNSP.

At the meeting convened in Madison, Wisconsin at which the Ashland pilot demonstration team (APDT) was formed (including representatives from WNSP, Newfields, David Crass (WNSP corporate attorney) and the DCI/DTI Team (TEAM)), Mr. Winslow of WNSP expressed his desire to investigate the applicability of the Process to contribute to the enhanced removal of coal tar present in the deep Copper Falls Aquifer. It was, therefore, decided that the project could be split into two parts that would embody the objectives of the USEPA (to validate the claims of the Technology) and yet speak to the concerns of WNSP. Thus, two demonstration areas were selected in separate areas of the site to meet the concerns of both parties.

One demonstration zone measuring approximately thirty (30) by fifty (50) feet (1,500 square feet), was located in the alley south of the maintenance building and positioned at the up-gradient end of both the shallow and deeper contaminant plumes. The area was roughly centered on the MW-15 monitoring well nest, and was selected to validate previous claims of the Technology (contaminant destruction, characterization & bioenhancement). A second area measuring forty (40) by sixty (60) feet (2,400 square feet), was located at the south side of St. Claire Street (including part of the courtyard) at the north side of the maintenance building. This area included the

MW-13 monitoring well nest as well as the three (3) operating extraction wells **EW-1**, **EW-2** & **EW-3**. This demonstration zone was selected to address the concerns of WNSP to evaluate the potential of the Technology to increase the efficiency of the coal tar extraction system in the deep Copper Falls Aquifer. The blue shaded area in **Figure 1** (attached) denotes the pilot demonstration area around **MW-15**, while the pink shaded area denotes the pilot area around **MW-13** and the extraction wells **E-1**, **E-2** and **E-3**.

The vertical treatment interval for the pilot demonstration in the **MW-15** area was divided into three (3) zones. The shallow zone, from ground surface to approximately fifteen (15) feet below ground surface (fbgs) was designed to treat contaminants in the Filled Ravine (a geological artifact extending in a curving manner from U. S. Highway 2 south of the site, through the site to the old shoreline of Lake Superior to the north). Underlying this treatment interval, from approximately fifteen (15) to twenty-five (25) fbgs, is a ten (10) foot layer of a hard clay aquitard known as the Miller Creek Formation. Underlying the Miller Creek Formation is the Copper Falls Aquifer. A treatment interval from approximately twenty-five (25) to forty (40) fbgs was selected to investigate the up-gradient extent of the coal tar contaminants in the Copper Falls Aquifer.

The QAPP design called for the injection to be performed from the bottom of the treatment interval (forty (40) fbgs) up through the Copper Falls formation to the bottom of the Miller Creek Aquitard (twenty-five (25) fbgs). At this point the Miller Creek was to be grouted up to the bottom of the Filled Ravine (fifteen (15) fbgs). The Filled Ravine was then to be treated to the surface. This injection sequence was to be conducted in a single placement of the injection tooling. The application was then to be repeated until all of the designed injection points (twenty-three (23) in all) had been completed. Prior to the implementation of the injection program, U.S. EPA and their SITE contractor (Tetra-Tech) were to collect soil and groundwater samples to develop baseline concentrations for the various contaminants.

The remedial design for the **MW-13** pilot area called for the injection of the *Cool-Ox*TM reagent from eighty (80) fbgs in the Copper Falls Aquifer to thirty (30) fbgs the bottom of the Miller Creek Aquitard. The injection points (twenty-seven (27) in all) were then to be grouted from thirty (30) fbgs to the surface to seal the Aquitard. This would prevent cross-contamination of the Copper Falls Aquifer and Filled Ravine. **Figure 1** depicts the areal locations of the pilot demonstration areas while **Figure 2** depicts the cross-sectional configuration of the vertical injection zones.

MW-15 Application and Findings:

Before the injection work began, problems arose that necessitated amendments to the QAPP. While implementing the work scope of the sampling protocol to establish base-line contaminant concentrations for the soils in the **MW-15** area, it was discovered that contamination in what was thought to be the Filled Ravine (FR) was virtually nonexistent in areas greater than ten (10) feet from the south wall of the maintenance building. This meant that the southern twenty (20) feet (two-thirds (2/3)) of the **MW-15** pilot demonstration area was for the most part free of contaminants. At this point, the

QAPP was amended and the majority of the samples were collected within the area ten (10) feet from the wall of the maintenance building. **Figure 3** depicts what was defined to be the free product in the shallow Filled Ravine and deeper Copper Falls Aquifer. **Figure 4** should be consulted for the approximate locations of the sampling points as they were ultimately located. Deeper samples revealed that coal tar residuals were present in the Copper Falls Aquifer in the pilot demonstration area. Sampling points were placed to develop data on the contamination of the Copper Falls Aquifer are also found on **Figure 4**. Analysis of the samples collected coincide with the consultants (Newfields) prior conclusions of contaminant locations as depicted in **Figure 3**.



Photo 1 above, taken in July 2006, depicts the **MW-15** pilot demonstration area. The dark depression at the base of the down spout to the left of the gas service (see arrow), reveals the location of the gas holder discovered during the injection activities in November. This area corresponds to the depression in the foundation of the men's lavatory just inside the wall.



Photo 2 depicts the site undergoing treatment. The green enclosure (see arrow) housed the *Deep-Shot Rig* to protect mixing equipment from the cold and snow.

Injections:

Initially, twenty-three (23) injection points (IPs) arranged on an eight (8) foot matrix were to be completed in the **MW-15** pilot demonstration area. As explained above, the vertical injection design for these IPs was to include injecting *Cool-Ox*TM reagents from the bottom-up, beginning at forty (40) fbgs (in the Copper Falls Aquifer) up to twenty-five (25) fbgs (the bottom of the Miller Creek Aquitard), then grouting up to fifteen (15) fbgs (the bottom of the Filled Ravine), then again injecting *Cool-Ox*TM reagents up to the asphalt surface and

grouting the upper two (2) feet with bentonite to seal the formation. The injection event was initiated with Deep Injection Point (**DIB-1**) (see **Figure 4**).

This IP location was selected because it was believed it was far enough west to include all three (3) geologic formations. However, from the surface, the injection was indicative of penetrating only very hard clay. Although the forty (40) fbg depth was achieved and the injection completed in the Copper Fall Aquifer,



As winter comes to northern Wisconsin **Photo 3** reveals the wisdom of providing heated cover for the pumping equipment.

retrieval of injection tooling was extremely difficult with the bottom ten feet of rod being lost. The IP was grouted from twenty-five (25) feet to the surface with Portland cement. It was the opinion of the experienced TEAM Direct Push Technology (DPT) rig operators, that there was no variation in the consistency of the upper twenty-five (25) feet and that the hard clay formation of the Miller Creek Aquitard was encountered at



The yellow arrow in **Photo 4** above depicts the location of **MW-16**. The orange arrow shows the location of the soil sampling point where the *Cool-Ox™* reagent was expelled to the surface. The unreacted white residue and the absence of odor indicate the absence of contaminant.

the surface. On-Site Environmental (OSE) was conducting sampling at the time and investigation of the core samples and discussions with the OSE operator led to the conclusion that the homogeneity of the upper twenty-five (25) feet of strata did not contain fill material indicative of backfill activity in the Filled Ravine. Because of these findings, all four-hundred eighty-five (485) gallons of reagent slated for this IP was injected in the twenty-five (25) to forty (40) foot interval. When the injection head was disconnected, expelled foam through the injection rod indicated the presence of contaminant. However, the

typical odor associated with coal tar was not observed. Instead, odor associated with reacted coal tar was noted. It was concluded that coal tar residuals were present in the Copper Falls Aquifer at this IP.

Deep Injection Points (**DIB-2**) and (**DIB-3**) were completed in the manner stated above with coal tar artifacts observed when the injection head was disconnected. It was concluded that coal tar residuals were present in the Copper Falls Aquifer at these IPs. However, when **DIB-3** was injected, reagent was reflected to the surface through **MW-16** and deep sampling point **SBS** (see **Figure 4**) located in the lawn just off the asphalt driveway to the southwest of **DIB-3**. Because the depth of **MW-16** was approximately fifteen (15) feet, it was concluded that a transmissive seam served as the transport mechanism for the reagent. This elevation coincided with the soil boring taken at the breakout point **SBS** in the lawn. In all three (3) of these IPs there was no indication of a heterogeneous strata usually associated with backfill activity.

At this point in the application work it became evident that the Filled Ravine was not located in any part of the pilot demonstration zone that had been treated. Conversations with the OSE personnel led to the belief that the Ravine was actually located at the far west extent of the proposed injection zone and perhaps out side of it entirely. In an attempt to define the exact location of the Ravine, TEAM member Kellogg conferred with the Newfields Project Manager, Dave Trainor and it was concluded that the Ravine may not be located in the demonstration zone. Since the contaminants in Filled Ravine were thought to be ideal for demonstrating the ability of the *Cool-Ox™* Technology to locate coal tar and stimulate bioremediation, another strategy had to be adopted.

As previously stated, the design of the QAPP for injections in the **MW-15** area called for an injection vertical in the Copper Falls Aquifer from forty (40) to twenty-five (25) fbg, then grouting through the Miller creek formation to fifteen (15) fbg, then treating to the surface in the Filled Ravine. With the discovery that the Ravine was located outside of the treatment zone a modification of the QAPP was necessary.



Photo 5 above depicts a contaminant source (**SIB-07**) (see **Figure 4**) just outside of the west wall of the holder. Although most of the soil around the holder appeared to be relatively clean, three of the IPs outside of the holder near **SIB-07** exhibited characteristics of significant contamination.

During the baseline sampling event, it was discovered that the shallow soils in more than approximately ten (10) feet south of the maintenance building wall, contained very low concentrations of contaminants. However, the area within the ten (10) foot line between the maintenance building wall and the gas main in the alley, contained high concentrations of contaminants. It was, therefore, mutually determined by the TEAM and USEPA representatives that the application could be divided into two work tasks. The first, would address the contaminants in the Copper Falls Aquifer by treating from forty (40) to twenty-five (25) fbgs as defined in the original design. However, the Miller Creek Aquitard would then be grouted with Portland cement, entirely to the surface. In a second work task, the vertical injection zone originally designed to address the Filled Ravine (fifteen (15) fbgs to ground surface), would be treated in a separate series of injections. The surface would then be sealed with bentonite grout.

The shallow application would concentrate on the area near the building instead of the entire demonstration zone, as was originally designed. It was believed that this modification would address the two vertical intervals in a manner that would speak to the original objectives of the QAPP. Namely, locating contaminants with the Process, facilitating their destruction and accelerating subsequent bioremediation. While this modification called for the placement of nearly double the injection points, the work could be accomplished in approximately the same amount of time as originally estimated because of the additional work necessary for the three phases of injecting into the Copper Falls Aquifer, grouting the Miller Creek, injecting the Filled Ravine and grouting the surface as was originally planned. The greatest time savings was in not having to clean-up the grout pump and flush injection hoses twice for each injection point.



Photo 6 (see arrow) above depicts another location (SIB-16) where a contaminant source was located.

Modifying the QAPP necessitated the redistribution of the IPs and *Cool-Ox*TM reagent to attempt to retain the objectives of the pilot demonstration. Because of the reactions observed in **DIBs-1, 2 and 3**, it was decided that the contamination in the Copper Falls Aquifer had intruded into the areas of these IPs. Therefore, it only remained to find out how much further to the south the plume extended. The TEAM concluded that this could be accomplished with the placement of fourteen (14) deep injection points. However, it was quite evident

that the concentration of contaminants and their location in the shallow (Miller Creek Aquitard) posed a challenge that required a more concentrated effort. Thus, it was decided to place twenty-five (25) IPs in a random pattern to treat the area where the baseline sampling event had located the majority of contaminants. The 11,110 gallons of reagent slated for the treatment of both zones was divided into approximately 7,500 gallons for the shallow zone and 3,600 gallons for the deeper Copper Falls Aquifer. The injection work began with the treatment of the shallow soil.

Treating the Miller Creek Aquitard

In effort to delineate the contaminant sources located by the baseline sampling in the shallow zone an IP (**SIB-01**) was placed approximately two (2) feet east of **MW-15** (see **Figure 4**). It was also reported that this well had produced free product. Evidence of coal tar on the probe rods during the initial stages of the work as they were extracted from the IP, indicated the presence of significant concentrations of contaminant. Heavy thick foam at the surface was another indicator. A second IP (**SIB-02**) was placed just south of the underground gas line in the alley. No indication of a contaminant source was observed, either by odor or foam. This coincided with the findings of the consultant and



Photo 7 above depicts the location of the holder wall (see flags) revealed by utilizing the probing technique of the DPT rig. The top of the wall was approximately eight (8) fbgs. The holder floor was found to be at eighteen (18) fbgs.

fbgs. While IPs to the south of the refusals demonstrated very little contaminant concentrations, those to the north were replete with coal tar. This discovery brought TEAM member Kellogg to the conclusion that the holder shown on the Sanborn Maps that was reported to have been removed, may in fact still be in place. This supposition was based upon the experience DCI has gained remediating numerous MGP sites and the norm of finding old plant artifacts that were suppose to have been removed. If the holder was there, the wall should be present in a semicircular pattern.

the baseline sampling event. However, monitoring well **MW-16** and sampling point **SBS** were influenced. Since the IP did not penetrate the Copper Falls Aquifer, it was assumed that a transmissive seam was present. After consultation with the SITE team, it was concluded that the majority of the shallow injections should be placed within an area between the maintenance wall and the underground gas line to the south.

As the injection work near the building commenced, several IPs encountered refusal at approximately eight (8) feet

To verify this possibility and attempt to locate the holder, a point was driven near an IP where refusal had been encountered at the eight (8) fbg elevation. When the probe again met refusal at the eight (8) foot level, the exercise was repeated at four (4) foot linear intervals until a semicircular arc was delineated. Based upon the type of reactions noted in **Photos 5** and **6**, it could be determined that with the exception the soil located to the west outside the holder, most of the area outside the holder was relatively clean. However, it appeared that the holder itself contained several feet (perhaps as much as six (6) to eight (8) feet) of coal tar. A discussion of the activity concerning treatment of the holder can be found below.



Photo 8 above depicts the location of the holder wall (see flags) from a west to east direction. The arrow indicates the location of the contaminants found outside the holder wall in the shallow (Miller Creek Aquitard). The remainder of the shallow pilot demonstration area outside the holder was relatively free of pollutants.

As stated earlier, the objectives of the pilot demonstration in the **MW-15** zone, were to determine if the Technology could:

1. actually locate contaminant sources,
2. reduce concentrations of contaminants by abiotic chemical oxidation and;
3. demonstrate that subsequent bio degradation was enhanced by the oxidation process.

Detecting Contaminant Sources: **Photos 5** and **6** graphically demonstrate that the Technology is reacting with contaminants and thus indicating that sources have been located. When these injection points are compared to other IPs such as **SIB-15**, **SIB-2**, and **SIB-9**, where prior sampling confirmed that little or no contamination was present and where no surface reactions for noted, it is evident that the Technology can detect contaminant sources, under the conditions present at this site. It should be pointed out that this feature is a qualitative technique that is useful in characterizing source areas at sites. To some degree, the experienced operator can determine if the sources contains free product or merely adsorbed contaminants. Although, the Process does not provide

the quantitative feature necessary to calculate the mass of contaminants present in the source, it does provide the consultant with a useful tool to conduct subsequent sampling events with more surgical precision than current random sampling procedures provide.

Fostering of Biologic Degradation: While the site work was on-going, the TEAM received a series of questions concerning on-site observations as well as the results of the January sampling event. These questions are quite relevant to understanding the application of the Technology and will be included in bold italics at appropriated places in the narrative.

TEAM member Tom Douglas reviewed the soil and groundwater data from the site with members of the Tetra-Tech and EPA SITE group and determined that many of the soil borings in the **MW-15** area were actually within the former holder. Because of the very large mass of coal tar present in the holder, it was difficult to determine the results of the Technology application on contaminant reductions or microbial proliferation inside that the vessel. However, observations made by Mr. Douglas at other sites where significant free product was encountered as well as at other MGP sites confirmed that the *Cool-Ox*TM application had a positive remedial effect in the holder. These phenomenon will be discussed later in this report.

Question: The HPC data indicates that bacterial population has increased in some locations, but again, the data are scattered, and there are no strong or clear overall trends.

Although the mass of coal tar in the holder certainly presented challenges to the conclusive analysis of the effect of the Technology on determining mass reductions and bioremedial impact, contaminated soils outside the holder provided an acceptable venue for these determinations. Comparison of samples taken outside the holder with those taken inside the vessel proved quite useful. The biologic and contaminant concentration data were evaluated specifically in two locations. These were borings **SB-12** and **SB-N** (see **Figure 4**). Soil boring **SB-12** was obviously within the former gas holder and this data proved very insightful. To evaluate and compare data, contaminated soil data from a location outside of the gas holder was selected. To accomplish this, the TEAM focused on soil boring **SB-N** because it was located in a contaminated area just west of the holder. This data also provided very useful results.

Soil and groundwater biologic and contaminant concentration data were tabulated and are presented on the attached **Charts 1 to 8**:

1. **Chart 1** shows that the 24 Hour Heterotrophic Plate Count dramatically increased in **MW-15** although the well was in the former gas holder and extremely high levels of contaminants were present in both the soil and groundwater. Counts increased from less than 10 MPN of CFU/ml to 93,200 between October 2006 and January 2007.

2. **Chart 2** has a similar trend for the Hydrocarbon Degraders detected in **MW-15**. Such counts went from less than 10 MPN of CFU/ml to 36 between October 2006 and January 2007. This provides further evidence that the treatment fostered or enhanced Hydrocarbon Degrading bacteria even when oxidation occurred and while very high contaminant concentrations were present. This is very significant and supports the findings of Cassidy's bench scale experiments to determine the effect of the *Cool-Ox*TM Technology on coal tar collected from other sites. Not only did the indigenous bacteria survive the initial oxidation they were able to multiply and proliferate, even under such harsh conditions. Several of Cassidy's papers have been attached as bench scale examples to corroborate these findings.
3. **Chart 3** provides more evidence to document that biologic enhancement occurred. The Total Plate Count in water from **MW-15** increased from 18 MPN of CFU/ml in October 2006 to 1,511 MPN of CFU/ml in January of 2007. Although only three (3) orders of magnitude, this demonstrated that indigenous bacteria were enhanced and biologic activity was fostered as a result of the *Cool-Ox*TM treatment. To some observers, this may not be significant however, again given the harsh environment, it is remarkable that the microbes survived at all.
4. **Charts 4 and 5** demonstrate that the levels of contaminants dissolved in the groundwater decreased significantly from October 2006 to January 2007. This decrease may have resulted from many mechanisms including the increased biologic activity at the site. A separate discussion on contaminant contaminations follows below.
5. **Chart 7** displays the results provided for soil boring **SB-12**, which was in the gas holder. The biologic data for the soil does not show dramatic increases for the parameter analyzed (24 and 48 hour THPC, HD, and TPC) as was noted in the groundwater. However, the soil results do indicate that the biologic activity was still present in the soil and that it had not been inhibited by the treatment. This was observed even though most of the contaminants were present in the soil and the chemical oxidants had obviously reacted strongly with the heavily contaminated soil. Therefore, it is believed that the data demonstrates that the biologic activity is still ongoing and the biota are viable and are contributing to the reduction of contaminants in the soil even under such extremely difficult conditions. As the mass of contaminants are converted oxidatively to less toxic and more biodegradable molecules, the biologic activity should be sustained and will likely increase even in this soil providing sufficient nutrients, moisture and oxygen are available.
6. **Chart 8** displays slightly more promising results for the biologic activity in the soil. It appears that subsurface conditions were more favorable for biologic activity in the area outside of the former gas holder after the *Cool-Ox*TM treatment was performed. This occurred between October 2006 and

January 2007. It appears that biologic activity increased following the treatment performed in the **SBN** area. A dramatic increase in the Total Heterotrophic Plate Count (THPC) was noted at a depth of nine (9) to ten (10) fbg. Significant increases were also noted at the eleven (11) to twelve (12) fbg and the thirteen (13) to (15) fbg sampling locations. The increases were so dramatic that the vertical axis (24 hour Heterotrophic Plate Count) had to be logarithmically presented. Based upon the historical bio-inactivity in this location it no doubt occurred because of the treatment performed in and around the holder.

Treatment of the MW-15 Holder

Photos 7 & 8 depict the position of the holder as determined by DPT probing. Subsequent to locating the vessel additional points were driven to determine the relative thickness of the wall. It was found that the wall was approximately one (1) to one and one half (1½) feet thick and extended from circa eight (8) to eighteen (18) fbg where the holder floor was encountered. No refusal of the type associated with striking concrete was noted, therefore, it appeared that the holder floor was composed of the same type of clay as is present in the Miller Creek Aquitard. This was later confirmed when samples taken from the bottom of the holder during the January and April sampling events, contained this type of soil. From an

east to west direction, the arc of the holder began approximately two (2) feet from the “L” (by the gas service) in the maintenance building and extended westward along the wall approximately twenty-four (24) feet. Bisecting the arc, the wall extended approximately eight (8) feet from the building wall at the furthestest point. The remainder of the holder is located under the maintenance building. **Figure 4** depicts the approximate location of the holder.



Photo 9 above reveals emulsified coal tar expelled to the surface after reaction with the Cool-Ox™ reagent. Note the physical appearance of the material has changed from the sticky, honey-like consistency to that more resembling “cake batter.” The edges of the “puddle” show folding and the “rough dull” surface feature is caused by the presence of reaction gasses. The temperature was indicative of groundwater. The slight bump in the middle (see arrow) reveals the IP (**SIB-14**) where the reaction is venting.

During the treatment of the **MW-15** area, it became apparent that the majority of the contaminants were located in the holder. Of the twenty-five (25) injection points placed in the **MW-15** treatment area, sixteen (16) were placed inside the holder. Although the team had treated other coal tar sites with the *Cool-Ox*TM Process, the volume of coal tar expelled to the surface during the application was by far the greatest at the Ashland site. Although odors and a minor degree of foaming had been noted during the treatment of the holder, it was not until injection at **IP-SIB-14** that anything resembling coal tar was expelled to the surface. **Table 1** may be consulted for a complete review of the treatment in the holder and the remainder of the **MW-15** injection area.

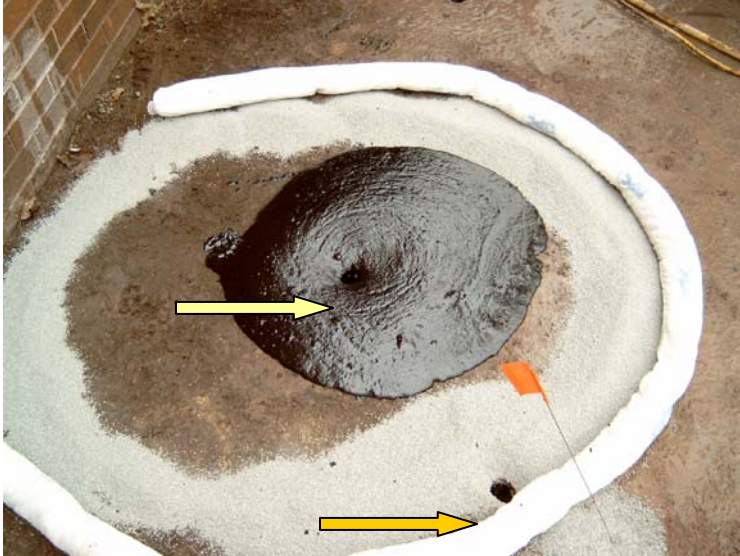


Photo 10 above depicts the reaction as it subsides and the emulsified coal tar runs back down the IP (see yellow arrow) as the reaction pressure subsides. Normally, the IPs are grouted after each injection is completed. However, (**SIB-3**) (see orange arrow) and some other IPs in the holder were left open to allow venting outside as a precaution because the majority of the holder was located beneath the maintenance building and the TEAM was concerned that vapors would intrude into the building.

Emulsification of Coal Tar

When coal tar was expelled to the surface through **IP-SIB-14**, it was an event that startled many of the TEAM members. Comments by all of the of the observers included such comments as "they had never seen coal tar flowable before." In fact, the coal tar that had been observed on DTP probe rods and on samples collected by OSE appeared as a sticky black substance that adhered to everything and was nearly impossible to remove. Conversely, the material percolating to the surface from the gas holder resembled previous coal tar samples in color only. The viscosity was no longer a thick honey like residue.

Rather a watery black flocculent substance that had lost its sticky adhesive characteristic and exhibited a much milder odor. Of course, it was no longer conventional coal tar. Instead, it had been emulsified by the action of the *Cool-Ox*TM Process. We are indebted to Paul MaCauley who had the quick wit to record the event as a video. Subsequent study of this video and comparison with films taken at other sites, aided greatly in revealing the emulsification Process. It also greatly influenced the chemical Process design applied to the **MW-13** pilot demonstrations area.

Because the pyrolysis process used to produce coal gas in the manufactured gas process, produces large quantities of aldehydes and phenolic derivatives such as creosote

and some types of polyaromatic hydrocarbons (PAHs), the coal tar is generally found to be acetic at a pH of six or less. The reaction of the coal tar with the Cool-Ox™ reagents, tends to raise the pH of the mass to eight. In addition, polyols or alcohols or other types of wetting agents referred to as surfactants are produced. Both of these actions produce an environment that causes the coal tar to convert to a more water soluble entity. The reaction gas, generally carbon dioxide contributes two vital functions. These are first, improved mixing as the gas is produced thereby opening up the treatment matrix, and secondly, decreasing the specific gravity of the organic mass as it is trapped in the matrix. All of these phenomenon contribute to the transformation of a thick sticky



Photo 11 shows the configuration of the emulsified coal tar (ECT) after the reaction has gone to completion and the surface is no longer impacted by subsurface activity. At this stage, the artifact is no longer coal tar but, instead a material that contains a large percentage of water (the transport medium) and reaction gasses. The material smears but, is not sticky or highly odorous like unreacted coal tar.



Photo 12 above shows TEAM Site Manager Rick Tolman collecting the ECT for drumming. At this stage, the ECT resembles “pudding” rather than the sticky, honey-like coal tar.

coal tar mass to an emulsified material which can be readily pumped and transported. The expulsion of this converted product to the surface has been previously observed and was expected at this site.

Question: John mentioned that the samples from the post-injection event were less viscous. It is unclear at this point if the reduction in viscosity is due to the Cool-Ox™ product or due to injection of large quantities of water.

No amount of water will facilitate dissolution or phase change of a material



Photo 13 depicts the ECT in a drum. Note the “rough” character of the surface indicative of the reagent reacting with contaminant and generating foam. At this point the physical characteristics of the original product are all but, gone.

that is insoluble in water at the normal groundwater temperatures encountered (see **Figure 7**). This can only be accomplished by actually changing the chemical characteristics of either the compound or the solution that is being used to dissolve or mobilize the compound. Chemically, this can be accomplished by several methods including the one used to emulsify the coal tar at this site. Other methods such as the use of surfactants can be used to increase the ability of the solvent (water) to carry the compound. The *Cool-Ox*TM

Process has used a combination of both physical and chemical phenomenon to increase the solubility and lower the viscosity and in short, convert the insoluble recalcitrant coal tar to a manageable product.

The question was raised during one of the telephone conference calls regarding surfactant production, if it might be prudent to “measure the dipole moment” of reacted samples to monitor the production of surfactants. While this procedure certainly can be used and is valid under laboratory circumstances, the procedure adopted to monitor surfactant production in samples collected from sites where



Photo 14 above shows the method used to contain reflections of ECT after the first material had been cleaned up and containerized. This method consisted of placing a cut section of a 55 gallon drum around the active IP and sealing the bottom (inside and outside) with bentonite granules. This effectively allowed the IP (see arrow) to vent while protecting the holder from intrusion by surface water. A drum cover was placed over the top to protect against rain or snow.

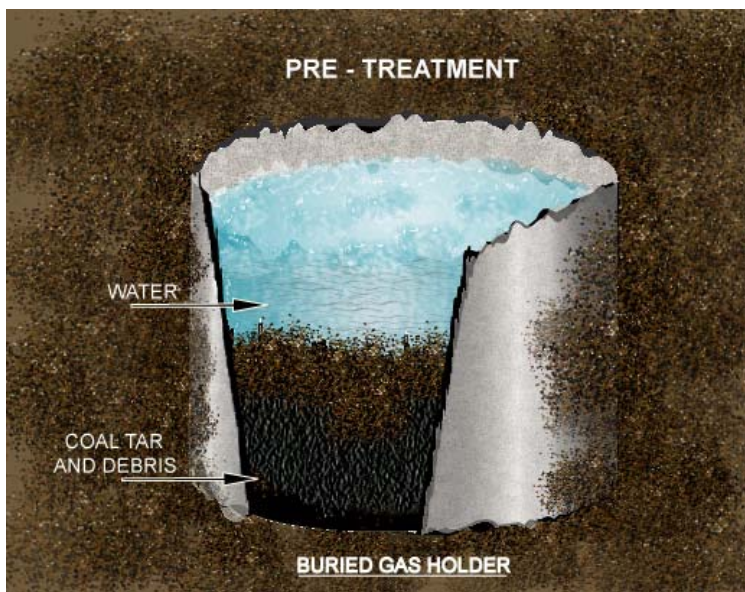


Illustration 1 depicts the suspected configuration of the coal tar/groundwater relationship prior to treatment with the *Cool-Ox*TM reagent. Note the water is phase separated from hydrophobic coal tar faction with the heavier tar collected near the lower portion of the vessel. During the injection activities as well as the sampling work, coal tar remnants were observed on the bottom eight (8) feet of the probe rods.

the groundwater may not be water-white, has been the measurement of surface tension. It has been shown that the surface tension of clean groundwater is approximately 70 dynes/cm. Laboratory experiments have revealed that pure water maintains this magnitude. However, the surface tension steadily decreases to a level of approximately 35 dynes/cm where the saturation point of the surfactants is it achieved. Groundwater samples collected from the site and submitted for analysis, maintained the 35 dynes/cm range consistently.

At this surface tension magnitude, the capacity of water to carry organic carbon contaminants (i.e., BTEX, PAHs, TPH, etc.) is greatly

increased. As micells are formed or long chain carbon molecules are held in suspension, the water will take on a milky or translucent appearance indicating its ability to transport large concentrations of normally insoluble compounds. This characteristic is central to converting groundwater to an extraction media and would not be possible without a wetting agent to bridge the gap between the hydrophilic water and the hydrophobic organics. This agent is referred to as a surfactant.

Contaminant Concentrations

When interpreting the data concerning contaminant concentrations where the data is derived from free product environments (in this case, emulsified coal tar in the holder), several factors must be considered. If one reviews the post treatment data in comparison to pretreatment concentrations of groundwater in **MW-15**, it would appear that substantial decreases of approximately 50% were achieved. This may be true. However, to obtain a clear understanding of the effect of the treatment on the contaminant one must not only consider groundwater concentrations but, take into account concentrations in soil as well. In this case, the data indicates that a six-hundred percent (600%) increase in contaminant concentrations in the soil. How can that be? Where did the product come from? The Process cannot manufacture the contaminants therefore, the answer lies elsewhere. The answer lies in the ability of the Process to emulsify and desorbed contaminants.

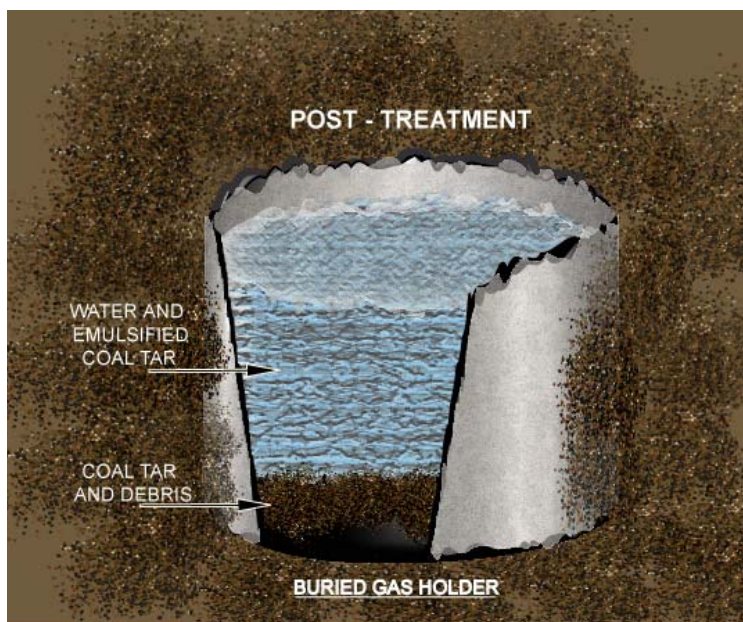


Illustration 2 shows the effect of the oxidation Process as it emulsified the coal tar thereby, increasing its solubility. This also causes the hydrophobic fraction to separate and migrate to the heavier molecular weight asphaltene like contaminants in the bottom of the holder. This phenomenon occurs when the concentration or quantity of oxidizer is stoichiometrically insufficient to convert the mass of contaminant to an oxidized byproduct.

As the reagent reacts with the contaminants adsorbed to the soil or cinder particles in the holder, it begins desorbing them over the extent of the contaminated vertical interval. Because the quantity of oxidizer is less than the amount required to oxidize the entire mass, the heavier hydrocarbons, as they desorb, separate to the bottom of the holder, where the organics are more soluble in the hydrocarbon mass than they are in the aqueous phase. Thus, rather than being spread over a larger vertical interval as is the case prior to treatment, they are concentrated in the hydrophobic mass at the bottom of the holder. This action is known as agglomeration (the concentration of a

hydrophobe with itself) and occurs when the quantity of surfactant is not sufficient to accomplish total emulsification (the breakup of a hydrophobe by surfactant action). In cases where the specific gravity of the organic hydrophobic constituent is greater than water, the material will desorb and sink. In cases where the specific gravity of the organic hydrophobic constituent is less than water, the material will desorb and float. This is not emulsification where the contaminants are finely divided into micells or molecular constituents. Instead, with agglomeration, the concentration of surfactant is just high enough to allow the contaminant to desorb and migrate as globules. When these globules meet, they combine to form a mass. This Technology is well understood by those schooled in the discipline.

Because this mass is more hydrophobic or oily in character, the hydrocarbon constituents such as BTEX or PAHs tend to be drawn into this hydrophobic environment. This action is no different than the simple laboratory exercise of using a separatory funnel to separate water soluble, components from oil soluble compounds. As the desorption occurs, the contaminants are concentrated in the hydrophobic mass at the bottom of the holder. Therefore, concentration of contaminants after treatment at the affected sampling elevation could be higher than before treatment if the reagent is not stoichiometrically matched to oxidize the target pollutants (converting them to hydroxylated organic

compounds wetting agents, i.e., surfactants) as well as the non-target heavier hydrocarbons. Based upon the soil samples collected from the bottom of the holder the target contaminants were concentrated in excess of six-hundred (600) times their pretreatment concentrations. However, investigation of the photos included in this report will reveal that, as expected, the integrity of the strata was disrupted by the treatment. Thus, accurate comparisons of pre-treatment contaminant concentrations drawn at certain depths against post-treatment sampling at the same depths may be impossible.

Question: The TIC data indicates absence of significant surfactant-type chemicals in the soil samples that can be observed above the hydrocarbon matrix. The presence of possible surfactant TICs (oxygenated and sulfonated organics) may have increased somewhat in the post-treatment groundwater samples. Are there any particular surfactants we should look for?



Photo 15 depicts a reflection of contaminant foam from **IP-SIB-16**. It should be noted that the reagent and a slight amount of contaminant foam can be seen surfacing at the building wall. This indicates that the reagent has reached the foundation and that contaminant was present at this location. This is the area west of the holder near the wall of the maintenance building where contaminant concentrations in the shallow soils were the greatest.

It may the have been appropriate to do EPA method 610 with GC/MS to identify the breakdown products from the large variety of baseline contaminants. More organic parameters are probably present than just those identified and listed on the spreadsheet. It has also been determined that when treating hydrocarbons of MGP sites it would be prudent to measure TOC and TRPH or TPH with fractionation. In this way, one could have documented the increases and decreases in TPH in specific contaminants. Thus, demonstrating that the contaminants are indeed being reduced to carbon based molecules with chains broken and very likely hydroxyl groups added

thereby, generating compounds such as alcohols, polyols, organic acids, etc. Keeping this in mind, it must be clearly understood that coal tar contains thousands of different chemical compounds, all of which are subject to oxidation. Therefore, it is nearly impossible to collect data on the individual reaction products produced.

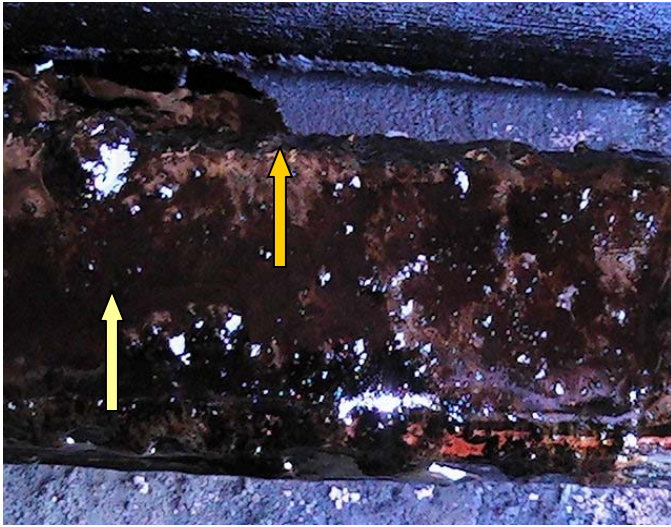


Photo 16 reveals the watery mass of ECT collected at the bottom of the holder during the January sampling event. Note the ECT (yellow arrow) and the soil and reagent mix (orange arrow). Samples collected at this interval prior to treatment were black with no phase separation

This challenge is greatly exacerbated by the dynamic nature of an on-going reaction where at one point in time, an analysis can reveal a broad spectrum of compounds. The problem is further confounded, if a second aliquot is collected from the same reaction vessel at a slightly later time. In this case, because the reaction is on-going, a completely different group of compounds may be present. Many of which may be daughter compounds of the reaction products found in the first analysis. This explanation is based upon laboratory experiments under controlled conditions. It is exponentially more complicated when the reactions are conducted under field conditions where it is nearly impossible to compend a complete list of all contaminants present.

Question: We have had difficulty in obtaining a sufficient volume of NAPL to conduct physical tests. The samples we have collected are black and oily, but appear to separate into thin layer of black material coating the sides and bottom of the sample bottles, with only a very thin layer of film at the top of the aqueous phase. Is this black granular NAPL (solidified NAPL) or soil particles coated with NAPL? Has Cool-Ox™ encountered any similar situations/sites where recovery of NAPL has been problematic?



Photo 17 above depicts the sampling interval use to collect the soil samples from the holder during the week of April 9th. The brown (clay) soil (see arrow) at the left of the photo is from the bottom of the holder.

It was also pointed out in one of the conference calls that when groundwater samples were received at the laboratory, the technicians were unable to conduct analysis because of the lack of sufficient product quantity.

The comment was made that the organic compounds were thin films that looked more like black dust adhered to the side of the sample vessels than what is normally seen in oily or hydrocarbon aliquots. This is not unusual and can be attribute to the fact that the solid oxidizers do not stop working as the samples are in transit. A measurement of the surface tension of such samples generally shows the surface tension to be somewhere in the mid to high thirty (30) dynes/cm range (seventy (70) is usual for groundwater not impacted with surfactants).

In many cases acid is used to “stabilize” the sample. This works by killing the microbes, where bio-samples are to be analyzed. However, when peroxygens are employed as hydrogen peroxide generators, the dissolution rate of these compounds is accelerated under acetic conditions. In the case of calcium peroxide, this reaction ($\text{CaO}_2 + \text{H}_2\text{O} \rightarrow \text{H}_2\text{O}_2 + \text{Ca(OH)}_2$) is well understood and it is easy to see that the calcium hydroxide formed by the reaction will quickly buffer the acid toward neutrality. A more subtle factor however, is the fact that the acid preservative, by increasing the solubility of the peroxygens, actually increases the availability of hydrogen peroxide over a much shorter timeframe thus, accelerating the oxidation of the organic contaminants in the sample. By the time the sample arrives at the laboratory, it does not represent the aliquot as it was collected and dispatched. One can argue that if this is the case, evolved gas should form a bubble in the container. This would be true only if the organics were mineralized to form carbon dioxide. However, since the bulk of the contaminants are hydroxylated rather than mineralized, any carbon dioxide formed could be of such low concentrations that they would reside at levels below their coefficient of solubility and thus, not be recognized as a gas phase in the sample container. The surfactants generated would also contribute to increasing the solubility. It should also be pointed out that all of the samples collected at the Ashland site contained soil or suspended soil. As such, the cation catalysts necessary for the formation of oxidizing radicals was inherent in the sample. Thus, the abiotic oxidation activity would proceed.

Question: If an un-sleeved injection point is used as a volunteer conduit to bring coal tar to the surface, isn't there a chance of contaminating more permeable layers (sand stringers, etc.) that might be present above the mass by intrusion of the coal tar into those areas?

Characterization vs. Remediation

The capability of the *Cool-Ox*TM Process to detect contaminant sources and reveal their locations by the reflection of “tell-tale” foam to the surface after the injection probe



is withdrawn has raised questions concerning contaminant recovery issues.

The primary question is, “if an un-sleeved injection point is used as a volunteer conduit to bring coal tar to the surface, isn’t there a chance of contaminating more permeable layers (sand stringers, etc.) that might be present above the mass by intrusion of the coal tar into those areas?”

Photo 18 above shows where a “soil” sample was collected during the January 2007 sampling event. This spill occurred when the soil sample collected from the bottom of the holder was so liquefied that the sampling tube was could not contain the soil sample. To retrieve soil samples, the probe was driven into the clay bottom of the holder to form a plug. The holder contents displayed the same watery characteristics during the April 2007 sampling event. This was evidence that the emulsion had not broken since the November treatment.

The possibility of that type of cross contamination is real and probably would occur. However, *one must remember that site characterization and subsequent remediation are two separate phases of the remedial program.* The *Cool-Ox*TM Process simply allows the work to progress from one phase to the other

without the need to waste time and expense locating contaminant sources. This is the qualitative step. In some cases, where (as is perhaps the case of MGP sites) it is suspected that massive quantities of contaminants are present, it may be prudent to conduct quantitative analysis to develop the information needed to select or design the proper remedial solution. It should also be remembered that during remediation activities, contamination of so-called clean soil or groundwater will occur.

To answer the question directly, based upon information gained at Ashland and similar sites, the *Cool-Ox*TM Process appears to produce the desired results for economically remediating contaminants found in deeper strata. Under normal circumstances, encountering a holder whether a gas holder or relief holder, during remediation of an MGP site, can significantly increase the expense of the job particularly if it contains coal tar. However, for the purposes of this pilot demonstration project it could not have been more welcome. The holder provided the opportunity to demonstrate in a closed vessel, the ability of the Technology to transform a heavy viscous material into a low viscosity material that could quite easily be extracted and

addressed under pro-forma pumping and materials handling techniques. If the mass of the contaminant can be extracted in this manner to the concentration level that will allow it to be a candidate for in-situ oxidation and combined bioremediation, the remedial costs can be greatly decreased. Other highly desirable qualities include eliminating the need to excavate thereby, eliminating the sensitive issue of odor emissions since all the work is completed without opening the earth.

To implement a remedial program to address this problem, it is believed that a series of extraction wells would be placed at strategic locations and elevations to aspirate contaminants as they are being desorbed by the Technology. Once the concentrations are reduced to the point that the reagents can treat the remaining contaminants, extractions can cease. This procedure will assure that strata above the affected zone are protected from cross-contamination caused by any off gassing from the Process. A detailed discussion of this Process or remedial design is beyond the scope of this report. However, the TEAM welcomes any questions that may be helpful in understanding the concept.

Question: *The groundwater samples from the thirty (30) day sampling event indicate that hydrogen peroxide was still present. Does Lundy have any idea what the initial concentrations should be? Mr. Lundy initially thought that the Cool-OxTM would not be present after about sixty (60) days, does Mr. Lundy still think so? Should Tetra*



Photo 19 above is additional evidence that of the accumulation of heavy coal tar artifacts at the bottom of the holder. It is expected that these samples will exhibit contaminant concentrations relative to those collected during the January 2007 event. Note the clay plug from the bottom of the holder to the right of the photograph (see arrow).

Tech delay the second post-injection event in order to give ample time for the Cool-Ox™ product to work and to record adequate percent reduction to support the primary goal of the project?

Sampling of the few groundwater monitoring wells at sixty (60) days would allow another snapshot of the dynamic conditions that are taking place at these few wells. Again it is important to add TRPH/TPH to the sampling regimen. The peroxide concentration should continue to decrease with time as these oxidants readily react with iron present in the soil and the organic molecules that are present at very high concentration levels. The slow continued generation of hydrogen peroxide is beneficial

as it improves the desorption of organic contaminants that were tightly bound to the soil and which had previously formed hydrophobic barriers to bacterial degradation.



Photo 20 shows a core sample taken outside the holder near **SBN**. Note the reddish color of the soil and the apparent absence of any coal tar artifacts. This core very different in appearance from those collected within the holder. It should be noted that the integrity of the soil allow meaningful samples to be collected from very defined vertical intervals. This was not possible in the holder where the heavy coal tar was desorbed from the soil, cinders (ash) and debris and settled to the bottom of the vessel.

The increased supply of oxygen will allow rapid aerobic degradation to occur until the amount of oxygen being generated and naturally diffused, decreases. Typically, it takes up to one-hundred (100) days for significant and sustained increases in biologic activity to be measurable. Many of the locations monitored showed acceptable levels of biologic growth after only a few weeks. This demonstrates that the *Cool-Ox™* did increase biologic activity in many points and that continued degradation of the contaminants of concern

is now taking place over a larger volume. In essence the treatment has made the site a larger bio-reactor.

The second sampling was delayed until the week of April 9, 2007. No samples were collected from the Copper Falls Aquifer. At the writing of this report, the sampling results were not completed.

During the injection work at the **MW-15** holder, the TEAM was faced with a wide variety of weather conditions, ranging from pleasant fall weather, to rain, to snow and freezing conditions. While the freezing weather made pumping and grouting difficult, these conditions were quickly overcome and subsequently the work proceeded without incident. The most troublesome condition, was rain which lasted for approximately two days with the water collecting in the low area atop the holder. Injection ports were grouted with bentonite to prevent casual water intrusion into the holder. Attempts were made to redirect the water from the roof downspout into the work area, however because water was also accumulating from the parking area, collecting the water using a drum vacuum prove to be the most efficient method of handling. The ground water was then tested and disposed of in an acceptable manner.



Photo 21 shows rain water that collected in the depression (see **Photo 1**) over the holder from the roof downspout and the parking lot. The water was contained in the treatment area by the boom (see yellow arrow) placed across the alley. The water was containerized with the drum vacuum (see orange arrow) and disposed of. The soil placed and compacted against the building wall to prohibit vapor intrusion into the men's lavatory in the building, where the foundation had settled can be seen (see blue arrow) at the right of the photo near the downspout.

Although when treating the holder, injection points were left open to vent any reaction gases, near the end of the injection work Photo Ionization Detection (PID) readings indicated that some intrusion was occurring in the men's lavatory inside the maintenance building. Prior to the initiation of injection work, it was noted that the foundation had settled over the holder area leaving a gap between the stud wall and the foundation. Being aware of this condition, the TEAM piled and tamped soil against the base of the maintenance building wall in an effort to seal off that area from the injection work. This strategy appeared to work well and it was not until near the end of the injection activities that higher PID readings were noted. Shortly thereafter, injection work in the holder was truncated.

Treating the Copper Falls Aquifer:

As previously noted, the injection work was initiated with Deep Injection Point (DIB-1) (see **Figure 4**). This IP location was selected because it was believed it was far enough west to include all three (3) geologic formations (Filled Ravine, Miller Creek Aquitard and Copper Falls Aquifer). However, from the surface, the injection was indicative of penetrating very hard clay. Although the forty (40) fbg's depth was achieved and the injection completed in the Copper Fall Aquifer, retrieval of injection tooling was extremely difficult with the bottom ten feet of rod being lost. The IP was grouted from twenty-five (25) feet to the surface with Portland cement. It was the opinion of the experienced TEAM Direct Push Technology (DPT) rig operators that there was no variation in the consistency of the upper twenty-five (25) feet and that the hard clay formation of the Miller Creek Aquitard was encountered at the surface.



Photo 22 depicts the reflection of coal tar from one of the deep injection points when the probe had been withdrawn. Because the pilot location was not impacted by the Filled Ravine, the threat of cross contamination between the Filled Ravine and Copper Falls Aquifer was greatly reduced. Thus, it was possible to observe contaminants reflected from the Aquifer. All IPs placed into the Copper Falls formation were grouted to the surface with portland Cement.

As stated previously, Deep Injection Points (DIB-2) and (DIB-3) were completed in the manner stated above with coal tar artifacts observed when the injection head was disconnected. It was concluded that coal tar residuals were present in the Copper Falls Aquifer at these IPs. However, when DIB-3 was injected, reagent was reflected to the surface through MW-16 and deep sampling point SBS (see **Figure 4**) installed by OSE located in the lawn just off the asphalt driveway to the southwest of DIB-3. Because the depth of MW-16 was approximately fifteen (15) feet, it was concluded that a transmissive seam served as the transport mechanism for the reagent.

This elevation coincided with sand seam artifacts found in the core sample taken at sampling point SBS at the edge of the lawn where the reagent broke out to the surface.

As stated above, based upon the findings during the initial work stages of the pilot demonstration, the QAPP was modified to divide the injection work into two phases. These were treating the shallow (Miller Creek Aquitard) in one work task and treating the deep (Copper Falls Aquifer) in a second. Initially, twenty-four (24) injection points (IPs) arranged on an eight (8) foot matrix were to be completed in the MW-15 pilot demonstration area. As explained above, the vertical injection design for these IPs was to include injecting *Cool-Ox*TM reagents from the bottom-up, beginning at forty (40) fbgs (in the Copper Falls Aquifer) up to twenty-five (25) fbgs (the bottom of the Miller Creek Aquitard), then grouting up to fifteen (15) fbgs (the bottom of the Filled Ravine), then again injecting *Cool-Ox*TM reagents to the asphalt surface and grouting the upper two (2) feet with bentonite to seal the formation.

Modifying the QAPP necessitated the redistribution of the IPs and *Cool-Ox*TM reagent to attempt to retain the goals of the pilot demonstration. Because of the reactions observed in **DIBs-1, 2 and 3**, it was decided that the contamination in the Copper Falls



Photo 23 depicts the foam caused by the reaction of the reagent with the contaminant, breaking out around monitoring well **MW-15B** as the reagent is injected at **IP-DIB-13**. Newfields had reported that they believed the integrity of the bentonite well seal had been compromised for some time by the corrosive activity of the coal tar. As the reaction occurred, the foam was contained by the cut drum. The monitoring well is protected by the PVC pipe extension. Expelled material outside the cut drum was collected for disposal and containerized in 17-H, 55 gallon drums.

Aquifer had intruded into the areas of these IPs. Therefore, it only remained to find out how much further to the south the plume extended. The TEAM concluded that this could be accomplished with the placement of fourteen (14) deep injection points. However, it was quite evident that the concentration of contaminants and their place in the shallow (Miller Creek Aquitard) posed a challenge that required a more concentrated effort.

Thus, it was decided to place twenty-five (25) IPs in a random pattern to treat the area where the baseline sampling event had located the majority of contaminants. The 11,110 gallons of reagent slated for the treatment of both zones was divided into

approximately 7,500 gallons for the shallow zone and 3,600 gallons for the deeper



Photo 24 reveals the proximity of **MW-15B** to the holder. Monitoring well **MW-15A** is located in the foreground. **MW-15A** was unaffected during the treatment. **IP-SIB-3** (in the holder) can be seen just behind the person in the blue jeans. Injection point **DIB-13** can be seen in front of the Geoprobe®.

Copper Falls Aquifer. After the revision of the QAPP, the injection work began with the treatment of the shallow soil at IP-SIB-1.

Upon completion of the injection work in the shallow zone, injections into the Copper Falls Aquifer were initiated with resumption at **DIB-4**. The objective of the injections into the Copper Falls Aquifer was to attempt to locate the southerly extent of contaminants that were present in concentrations high enough to be defined as a source area (free product or smeared) based upon the reaction of the coal tar with the *Cool-Ox*™ reagent. It

should be pointed out that under the original QAPP, this work was to be conducted to; a) locate the areal extent of the contaminant in the Filled Ravine; and, b) collect microbial samples to determine if the Technology did indeed contribute to the proliferation of intrinsic species. However, discovery that, in this area, the Miller Creek Aquitard extend from near surface to the Copper Falls Aquifer at circa twenty-five (25) feet, it was decided to investigate the contaminant range in the latter.

The results of the reactions coincide with the finding of Newfields as shown in **Figure 3**. Heavy reactions with foam and odors were noted to the west and southwest of the holder with milder reactions to the south and southeast. Only slight foam reactions



Photo 25 depicts the continuing reaction (note rising foam caused by the emission of carbon dioxide) after injection has ceased at monitoring well **MW-13B** just outside the holder. **IP-SIB-3** inside the holder can be seen at the top right of the photo.

were noted in **DIB-4** with no odor observed. To the south near **MW-16**, **DIB-7** exhibited a slight foam reaction with mild odor indicating the presence of low levels of contaminant. No reactions or odor was observed in **DIB-8** to the south however, communication with **DIB-7** exhibiting a slight amount of foam was noted during the injection at **DIB-8**. It was mutually decided by the TEAM and USEPA Group that further investigations to the south of **DIB-8** were not necessary.

During the injection at **DIB-13**, the bentonite seal at **MW-15B** was compromised. It is assumed that a combination of gases generated by reaction of the reagent with contaminant coupled with the hydraulic pressure of the injection caused this phenomenon. Under normal circumstances, if monitoring wells are properly installed, this does not occur. Upon observing that the bentonite seal been expelled by the reaction, Mr. McCollogh, New Fields Site Manager, indicated that Newfields had arrived at the conclusion sometime prior to the implementation of the pilot demonstration that bentonite around the base of the well had been compromised by the action of the coal tar residue. **MW-15B** is the deep well, screened from approximately forty-seven (47) to fifty-three (53) fbg.

Although, **MW-15A** is a shallower well, screened from approximately thirty-five (35) to forty (40) fbg, no appearance of well seal compromise was noted at that location. Because reaction gases tend to rise through the formation it would have been logical that **MW- 15A** should have been compromised rather than **MW-15B** if the latter had been in good repair. Since this did not happen, it can be assumed that the well seal around **MW-15A** is intact and that Newfields assumptions concerning the previous problems with **MW-15B** are correct. It should be noted that although the well seal outside of the well casing failed, no indication of increased elevation of the groundwater inside the well casing was observed. Therefore, it can be assumed that the strata below the forty (40) fbg injection interval, was not affected.

When it was observed that the bentonite seal of **MW-15** be was compromised, an extension was immediately placed on the monitoring well to avoid contamination of the Copper Falls Aquifer from the injection activity above. At the same time, a cut drum was placed around the monitoring well to contain expelled material. All material was containerized and segregated for proper disposal. Injection at **DIB-13** was ceased and the IP grouted with Portland cement as were all injection points.

A similar breakout at **MW-15B** be was again observed during injections at **DIB-14**. The same procedures were used to contain the reaction and collect the expelled material as before and injection of the Copper Falls Aquifer in the **MW-15** pilot demonstration zone was completed. The gas holder was a god-send field laboratory.

MW-15 Summary:

Findings during the injection work at the **MW-15** Pilot Demonstration Area that presented several unanticipated challenges, necessitated several modifications in the

Quality Assurance Project Plan (QAPP). These modifications began with discoveries made during the baseline sampling events and continued on through the finish of the injection work. The major findings and point of interest were as follows.

Filled Ravine: Although shown on the Sanborn Maps to be present in the **MW-15** pilot area, it was determined that this geological formation was in fact, located west of demonstration area. This presented significant problems because it was anticipated during project design that the Filled Ravine could contribute to the verification that the *Cool-Ox*TM Technology could locate contaminants, destroy them through in-situ chemical oxidation and initiate accelerated bioremediation. Fortunately, subsequent discoveries were made that supported these claims that did not rely on the Ravine.

Gas/Relief Holder: Although the historical records indicated that the holder had been removed, the injection work revealed that it was indeed, in place. When the south wall of the holder was located, it revealed that the majority of the holder was located under the maintenance building. Photographs taken during July 2006, indicate a depression of the pavement in the alley where the holder was located. Although unexpected, the presence of the holder provided a very positive opportunity to gather and verify characteristics of the Technology that would not have been possible had the holder not been present. Because the holder contained a significant volume of free product, the TEAM was able to demonstrate that the *Cool-Ox*TM Process could emulsify coal tar. Additional information was developed however, the ability to emulsified coal tar in large concentrations thus, breaking the viscosity and rendering it extractable by conventional means, opens the door to new remedial opportunities.

Bioremediation: Two locations were examined to determine the effect of the Technology on accelerating intrinsic bioactivity. One, from inside the holder and one located outside the holder. While the point inside the holder (**SIB-12**), demonstrated only moderate increases in microbial proliferation, it was concluded that because of the harsh environment, the fact that the bacteria survived and demonstrated some increase was significant. Conversely, the point outside the holder that was studied (**SBN**) revealed significant increases in microbial plate counts. The relationship between these two points demonstrated that in the presence of free product or highly elevated concentrations of contaminant, bioremediation will not make a significant contribution to site remediation. On the other hand, where contaminants reside primarily in groundwater or in concentrations below the toxic threshold of the microbes, bioremediation can be significantly accelerated by the Technology and provide the “finishing” (mitigation of contaminants subjected to abiotic chemical oxidation) step in cleaning up sites.

Contaminant Destruction Through Chemical Oxidation: As in bioremediation, the ability of the Technology to destroy contaminants by chemical oxidation must be investigated from two perspectives. Outside the holder where contaminant concentrations were low enough to be measured without the influence of free product, the reductions in target pollutants were very satisfactory and comparable to other sites treated where PAHs and BTEX were the subject of mitigation. However, in

the holder where free product was encountered the mass of contaminants was so concentrated that reductions, given the quantity of reagent injected, were impossible to measure. Therefore, an attempt was made to determine the effect of the reagent on contaminant destruction by analyzing for oxidation products. These were referred to as TICs. Unfortunately, at the writing of this report, the measurement of TICs is still under discussion. However, the coal tar in the holder underwent a change in physical character. This physiochemical change was a transition of the coal tar from a sticky, honey-like odorous material to an emulsified product that was to some degree water soluble or miscible. This transformation could not have occurred without the formation of surfactants. It has been shown that hydrocarbons are converted to surfactants by chemical oxidation. Thus, compounds comprising the make-up of coal tar were oxidized to form surfactants. In short, a portion of the mass of coal tar was oxidized. The question remains, what percent of the total mass was converted. Because the amount of contaminant was so great, the degree to which it can be measured remains to be determined.

Analytical results from samples taken within the holder show a fifty percent (50%) decrease in the concentration of target pollutants in the groundwater. While these analytical results are undisputed, one must remember that at the same time, concentrations of contaminants in the soil at the bottom of the holder increased by factor of six-hundred percent (600%). As explained in the text, these two phenomena are the result of a combination of chemical oxidation and surfactant production causing an agglomeration or concentrating of the contaminants at the bottom of the holder. While this was expected, it should be remembered that desorption of coal tar over the vertical treatment interval that may indicate a reduction in the concentration, may be misleading and may not be attributable to destruction under chemical oxidation. Rather, in this case where the reactions were occurring in free product, it is more likely that the agglomeration process concentrated the contaminant at the bottom of the holder where analysis indicated concentrations has increased. This is very different from the reactions observed in the **MW-13** treatment over where the lighter gravity material was “lifted” by the action of the oxidizer.

Emulsification of Coal Tar: Although the ability of the *Cool-Ox*TM Process to emulsify coal tar has been demonstrated elsewhere, the usefulness of this phenomenon was graphically demonstrated by the conversion of large quantities of this material in a confined unit (the holder). By converting the insoluble sticky, viscous mass of coal tar to a material (emulsified coal tar ECT) that is pumpable or extractable using conventional remedial tools, it has been demonstrated that new, very economical methods of site remediation are possible. This is by far the most important finding of this site demonstration.

MW-13 Application and Findings:

The objective of the pilot demonstration in the **MW-13** monitoring well nest, was to determine if the *Cool-Ox*TM Process could improve the efficiency of the extraction wells by increasing the desorption of coal tar contaminants in the Copper Falls Aquifer. This was to be evaluated by the increased volume of contaminants (against time) recovered in the wastewater treatment plant.

The remedial design for the **MW-13** pilot area called for a forty (40) by sixty (60) foot area (2,400 sq. ft.) to be injected with the *Cool-Ox*TM reagent. A vertical injection interval from eighty (80) fbgs in the Copper Falls Aquifer (approximately seven (7) feet below the bottom of the contaminant plume), up to thirty (30) fbgs (where it interfaced



Photo 26 shows the **MW-13** pilot demonstration area prior to treatment in early July 2006. The telephone service near extraction well **E-1** is located to the left of the photo (see arrow).

with the bottom of the Miller Creek Aquitard), was designed. This interval was chosen to assure that the entire vertical extent of the plume would be contacted by the reagent. The injection points (twenty-seven (27) in all) were then to be grouted with Portland cement from thirty (30) fbgs to the surface to seal the Aquitard. This would prevent cross contamination of the Copper Falls Aquifer with the Filled Ravine. **Figure 1** depicts the areal locations of the pilot demonstration while **Figure 2** shows the cross sectional configuration of the vertical injection zones.

While the spacing of the injection points was designed on a nine and one-half (9 ½) foot matrix, the discovery of structures and utilities in the injection zone during the actual application, necessitated the redeployment of the injection points such that, they would not impact of these interferences (see **Figure 6**). Note the green dots on the figure depict the location of the designed IPs, while the black numbers connote the location of the actual IPs as they were installed. Although the remedial design called for the injection of 33,633 gallons of reagent equally into the twenty-seven (27) IPs, the combination of utility interference, refusal from debris in the Filled Ravine and the encounters with the rock and hardpan in the Copper Falls Aquifer, rendered it impossible to adhere to the original design. **Table 3** contains a breakdown of each injection point including, the depth, the volume of reagent injected, obstructions encountered, and any reactions noted.

Although the entire volume of reagent was successfully injected, because of refusals encountered in the Copper Falls Aquifer due to the presence of cobbles and apparently hardpan seams, the average injection depth was sixty-three and one-half (63½) feet. **Figure 2** indicates that the depth of the contaminant plume is approximately seventy-three (73) feet. Therefore, if one calculates the vertical injection interval from thirty (30) to sixty-three and one-half (63½) feet, approximately seventy-eight percent (78%) of the volume of the plume in the treatment area was addressed.



Winter arrived prior to the start of work in the **MW-13** pilot area. **Photo 27** above depicts the area after the exclusion zone has been established. The red decontamination trailer is visible in the background.

At the onset of the injection work, it was noted that although the remedial design called for injection from the bottom of the Copper Falls Aquifer up to the Miller Creek Aquitard (a bottom up method), it was thought to be prudent to place the first injection point using a top down method. This change was incorporated with the consent of the EPA SITE team to assure that any reaction gases evolved from the reaction of the reagent with the contaminant would be vented to the shallow extraction well **E-1**. **IP-1** reached the entire eighty (80) foot vertical

interval. Approximately three-hundred (300) gallons of the 1,200 gallons design for the injection point was placed in the top down injection method. The remaining nine-hundred (900) gallons were to be injected using the bottom up protocol. However, the pressure activated injection tip plugged and the rods had to be extracted and the injection tip freed. Upon extraction, it was discovered that the bottom fifteen (15) feet of tooling including the injection tip was lost. The rods were fixed with an expendable point and again driven into the **IP-1** injection port. However, refusal was encountered at the sixty-five (65) foot level. It was believed that the replacement rods might have contacted the fifteen (15) feet of rods lost in the prior activity. It is important to note at this point, that the depth to groundwater in **E-1** was of approximately nineteen (19) to twenty (20) fbg. It was determined during the probing process that the bottom of the Filled Ravine in the **MW-13** pilot area was approximately fifteen (15) fbg. Therefore, no upwelling of groundwater from the Copper Falls Aquifer was expected. During the entire application process, interference or refusal from contact with cobbles in the Copper Falls Aquifer was encountered at every injection point.

No reactions in **E-1** or any of the **MW-13** monitoring wells were observed during the injection of **IP-1**. However, a volunteer breakout was noted near the telephone service approximately ten (10) feet east of the injection point. Injection was halted and the expelled material contained and containerized for disposal. The breakout was plugged with bentonite and an additional two-hundred (200) gallons of reagent injected. **IP-1** was completed and grouted. **IP-2** was placed approximately three (3) feet north of **E-1**. After injecting approximately six-hundred (600) gallons, thick reaction foam again broke out by the telephone service. **IP-2** was abandoned with the intention of moving to the south end of the treatment area. However, when the probe rods were withdrawn from **IP-2**, it was observed that Groundwater had risen to the top of casing in extraction well **E-3**.

Extraction well **E-3** is located approximately forty (40) feet across the treatment area to the south. This was an event that the TEAM had hoped to observe at extraction well **E-1**. Investigation of **Figure 2** will reveal a considerable upward gradient at the bottom of the Miller Creek Aquitard between extraction wells **E-1** and **E-3**. Reaction gases generated from the injection activities near **E-1** had evidently migrated upward to extraction well **E-3**. This type of communication within the Copper Falls Aquifer was very welcome. It demonstrated that reaction gases could be used to desorb and lift contaminants in the deep strata.



Photo 28 above shows the green shelter housing the “Deep-Shot Rig.” *Cool-Ox*TM reagents are secured in the truck at right. This vehicle is locked at night, as a safety feature, to prohibit vandalism or access by only the curious. The crew can be seen readying the Geoprobe[®] and the Skid Steer for the days work.

When treating heavily contaminated service station sites, once a breakout occurs it is typical that most other injection points will communicate with the breakout point. It has been discovered that the process typically follows the contaminated strata (smear zone) where reaction gases create a migration path for the reagent. It has been demonstrated that if the application is applied in a doughnut pattern with concentric points being placed ever further way from the breakout point, a transmissive condition is set up in the subsurface and the distribution of the reagent is improved. With this in mind, the third injection point was placed near extraction well **E-3**.

It should be noted that extraction wells **E-1** and **E-3** were screened such that, the top of the screened intervals was near the bottom of the Miller Creek Aquitard. Because of the reaction gases, generated when the reagent contacts the contaminant, the process tends to lift contaminants. Because the Copper Falls Aquifer is composed of gravelly soils, it was believed that the upward direction of the process assisted by the artesian affect of the aquifer should lift any desorbed contaminants upward toward the extraction wells so that the freed contaminant could be captured by the extraction pumps.

As the injection work was implemented in the area of the **E-3** well, it was noted



The maintenance building sheltered the work in the **MW-15** pilot area from the fierce north winds off Lake Superior. However, the **MW-13** area was directly exposed to the numbing cold. The blue tarps shown in **Photo 29** above provided relief from the wind as well as privacy for the injection work.

that the rolling action of the reaction gasses expelled oily debris from the well casing. pH readings were collected when the groundwater had reached the top of the casing and it was noted that these readings were acidic near pH 5.5 to six (6). This would indicate that the reagent had not yet reached the top of the casing. As the groundwater continued to be expelled from the **E-3** casing, the pH slowly increased to eight (8) and the color of the groundwater turned to a milky dark tan. This indicated that the *Cool-Ox*TM reagent had reached the well and that surfactants were being formed from the contaminants. This phenomenon aids in the desorption of contaminants from the soil.

The mechanism of surfactants aiding in the dissolution of hydrophobic materials and in the formation of aqueous emulsions is well understood and will not be discussed further in this section of the report. Rather, the question here is; does the *Cool-Ox*TM Process oxidize hydrocarbon contaminants to produce surfactants from these types of pollutants? If the process can produce wetting agents (surfactants), it will contribute significantly to the efficiency of the pump and treat system in place at this site.

Unlike the phenomenon occurring in the **MW-15** holder (where free product is being desorbed from the soil and agglomerated at the bottom of the holder), the contaminants present in the Copper Falls Aquifer appeared to be much more dilute and of

a lighter specific gravity. This would be normal, considering the high



The torpedo heater in **Photo 30** above helps keep pump lines thawed and personnel to cope with the cold as they work in the vault at extraction well **E-1**.

concentrations of benzene in the contaminant mix (benzene will act as a solvent in coal tar hydrophobes in the same manner that gasoline acts as a diluent in oil, this lowers the viscosity and the specific gravity of the resultant solution).

The oxidation affect on these compounds (that are important to this pilot demonstration) can cause the physical characteristics to manifest themselves as a moiety in an aqueous media in at least three ways. These are as:

1. wholly dissolved hydroxylated (surfactants) molecules that are present as individual entities suspended in the water;
2. micells, minute particles of unreacted contaminants held together by the action of produced surfactants (micells give a milky appearance to the groundwater) and;
3. globules of product much larger than micells that are suspended in the groundwater because of their low (or near water) specific gravity.

Prior to treatment of the Copper Falls Aquifer, all contaminants extracted by the wastewater treatment system were in the form of globules. Therefore, the pilot demonstration evaluation criteria was based upon the increase in coal tar (in the form of globules) that could be separated and measured in the water treatment facility. Unfortunately, no protocol was developed to measure the presence of dissolved contaminants or micells which would pass through the skimmer tank undetected. The volume of these undetected contaminants could easily be much greater than those collected as free product. Because of this phenomenon, the condition of the skimmer tank in the water treatment plant was checked periodically for variations in pH and the appearance of the water as well as the contaminant.

Because no reactions were observed in extraction well **E-1** during injections in that area, it believed that the well screen may be occluded to the passage of all but, small volumes of water. Because of the low pump rate of the extraction pump, this fact

would not have been detected under normal operating conditions. Because the top of the well screen in **E-2** was least twenty (20) feet below the bottom of the Miller Creek Aquitard and since the reaction gas had caused the groundwater level in **E-3** to elevate to the top of the well casing, it was concluded that the majority of the groundwater affected by the process, would be collected from extraction well **E-3**. Therefore, when the reactions in **E-3** were observed, the pH of the water as well as visual observations of the water conditions in the water treatment plant separator were conducted. As the pH of the groundwater in **E-3** rose to eight (the optimum for the process) and the milky appearance (indicative of contaminant presence) was noted, the TEAM was alarmed that no similar changes were observed in the skimmer. This gave rise to the question of whether the **E-3** well was operable. Isolation of the well by service personnel indicated that it was not. Injection work was halted and the well pulled for inspection.



Photo 31 depicts the interior of the green shelter, employed to protect the *Deep-Shot Rig*TM from the freezing conditions outside. Propane heaters kept pumps and hoses from freezing. The ramp to truck storing the Cool-OxTM reagents can be seen at the left of the photo.

Investigation indicated that the pump mechanism in the well had been lost. A new pump was installed, tested, and when satisfactorily operating, the injection work was resumed. Shortly after the new pump was installed in **E-3**, the appearance of the groundwater in the separator became milky, the pH rose to eight (8), and the appearance of the contaminant on the surface of the water had changed from a smooth oily shine to a mottled, dull form similar to that of the contaminant material seen in the photos of the **E-3** vault.

Because the primary interest in the groundwater in the **MW-13** pilot area, was in contaminant mass extracted rather than analysis of contaminant species, no groundwater samples were collected for chemical analysis. Therefore, the effect of the technology on oxidation of individual chemical species was not investigated. This was a very unfortunate circumstance because such analysis might shed light on the effect of the application on oxidation of BTEX and PAH concentrations. Samples were however, collected by the TEAM to determine surface tension. As in similar MGP applications, it was found that the surface tension of groundwater collected from extraction well **E-3** had



Photo 32 above depicts only a few of the confrontations between the injection probes and the rocks in the Copper Falls Aquifer. The item in the middle is a new, undamaged expendable point. This provides a driving tip for the probe rods. When the probe reaches the desired depth, it is withdrawn two (2) to six (6) inches and the point pushed out by the pressure of the reagent. This initiates the injection process. Expendable points are used when the injection protocol calls for injections to start at the bottom of the vertical injection interval (VII) and distributes the reagent to the top as the probe is withdrawn. This is referred to as bottom-up (BU) delivery. The two items on each end are solid drive points that provide a drive point for retrievable tooling such as the Pressure Activated Valve (PAV), employed when the reagent is distributed from the top of the VI downward. This is called top-down (TD) delivery. All four (4) of the used point exhibit evidence of striking rocks.

dropped from approximately seventy (70) to thirty-five (35) dynes/cm. This would indicate that during the application, surfactants were produced and the ability of the groundwater to carry and transport dissolve phase contaminants was greatly enhanced. It should be noted, that although it has been demonstrated that surfactants are produced from organic compounds, including coal tar constituents, only relative terms rather than precise quantities or concentrations have been used (in this report) to attempt to describe the quantity or load of contaminants that can be transported by a given volume of water. This is because prior to the implementation of the site work, no methodology was developed or had been adopted, for the project to measure the precise contaminant or surfactant concentrations in terms of weight per unit volume (i.e., grams/Liter). Instead, the change in surface tension was employed to determine if surfactants were being produced.

A point should be made about the quality or type of surfactants produce by the *Cool-Ox*TM oxidation reaction. The term surfactant is an acronym for the phrase “surface acting agent.” The term is also used interchangeably in the chemical industry, with the phrase “wetting agent.” For the purposes of this report, this group of molecules are generally composed of an organic hydrocarbon (hydrophobic) molecule with a hydroxyl (hydrophilic) group added by the oxidation reaction. In their primary state, these molecules align themselves between groundwater (hydrophilic) and the coal tar

(hydrophobic) contaminant thus, “breaking up” the coal tar mass and allowing groundwater to transport significant quantities of the unreacted coal tar.

During the injection work, a breakout occurred outside the casing in the **E-3** vault. The appearance of this effluent was substantially different than the contaminant material expelled from inside the casing. Instead of the much darker frothy ECT, the material from outside the casing appeared as a thick brown foam. This was collected and containerized in drums. Photos of the material in the vaults as well as in drums are included in this report. This effluent closely resembled the foam ejected from the breakout point near the telephone service near **E-1**. It should be noted that as the material was collected by the vacuum equipment and containerized, the oxidation reactions continued to proceed. When the effluent was collected from the **E-3** vault, a large void was discovered containing wooden debris and ash. It was assumed that



Photo 33 above shows a close-up of an expendable point on the right that has been driven into the point holder with such force that the two parts appear to be welded together. This point was retrieved from **IP-7** near extraction well **E-2**. It can be seen that the point was “peened” on all sides by striking rock. Pump pressure applied at 1,200 psi could not dislodge the point. Prior to the initiation of the injection work, it was believed that the hard clay structure of the Miller Creek Aquitard would provide the greatest test for the driller. It turned out that the cobbles in the Copper Falls Aquifer provided the real challenge. Refusal proved to be so severe that the average depth of penetration was 63.5 feet.

this material was part of the fill in the Filled Ravine. Approximately one and one half (1½) cubic yards of sand was required to fill the void. This event prompted the TEAM to review the coal tar extraction options that it had evaluated prior to implementing the injection work in the **MW-13** pilot demonstration area.

As the injection work proceeded in the south part of the treatment area, approximately 6,000 gallons of reagent had been injected with no observations in any of the wells or any breakout point. However, while injecting at **IP-18**, breakout occurred near the foundation at the north side of the maintenance building. Photographs of this event are included in the report. This breakout was approximately eighty (80) feet from the injection point. The effluent was quickly collected and containerized and the area



Photo 34 above depicts the volunteer breakout (see orange arrow) near the telephone service (see yellow arrow) where the contaminant reacting with the *Cool-Ox*TM reagent was expelled to the surface through a previous sampling point. This occurred during injection in **IP-1** near extraction well **E-1**. Note, the reaction was contained using the cut drum method that proved successful during the injection work in the **MW-15** pilot area.

washed down with clean water. The clean up water was also containerized for proper disposal. During the cleanup work it was noted that the foundation had apparently settled in the area of the breakout. The sidewalk had also settled at the side near the building and separation of the bricks in the wall had previously been tuck-pointed. Noting that a similar settling of the soil in the **MW-15** pilot area had occurred over that holder prior to the pilot demonstration project, the question arose, could the large holder shown on the Sanborn Maps as being located in this area, be present? If so, like the **MW-15** holder, it could contain large quantities of coal tar which could continue to be an on-going contaminant source.

The question of artifacts from the old MGP operation remaining in place, was raised by other events that occurred during the injection operation. While injecting at **IP-10**, a clear liquid with a specific gravity lighter than water, was reflected to the surface during the grouting operation. The material had the odor and appearance of diesel fuel or kerosene. The TEAM believes its source is the old naphtha tank, shown on the Sanborn Maps as being located in that area. This event, coupled with the observations made during the entire **MW-13** injection program, gives rise to the supposition that, in the immediate area of the old MGP operations, the Miller Creek Aquitard may not be as impermeable as previously believed. How did the deep aquifer get contaminated?

A critical point concerning the integrity of the Aquitard is based on the premise, that all studies and data collected at the site prior to the *Cool-Ox*TM application, were based upon the supposition that (with the exception of the artesian affect of the Copper Falls Aquifer), the pressures exerted by the surface water or shallow groundwater in the Filled Ravine; were downward. The generation of reaction gases from the Technology, would reverse the direction of the pressure gradient so that the pressure would be upward from the injections in the Copper Falls Aquifer. It should also be noted that gases are more intrusive at negotiating strata than liquids particularly, viscous liquids such as those

produced by MGP operations. Because of its weight and settling properties, the TEAM selected Portland cement rather than bentonite as a grouting material. Although the possibility exists that some of the injection points may have been compromised, the volume of reagent and groundwater reflected up into the Filled Ravine would indicate other pathways to be present also.



Photo 35 shows the drum vacuum employed to capture reacted contaminants and reagents that were expelled to the surface during the application process. Extraction well **E-1** can be seen in the background (see arrow).

To access the area under St. Claire Street, it was necessary to probe at an angle. This method was employed because the city preferred not to have the street breached. The angle probing did not present a problem and delivery of the reagent was accomplished. During the injection process contaminated groundwater and reagent was reflected to the surface through monitoring well **MW-13**. This was an important event from several perspectives. Most importantly, the initial expulsion of effluent contained very high concentrations of emulsified coal tar. This material had no

doubt, collected in the well screen around the PVC casing of the well itself. The contaminant was aspirated from the well and contained for disposal. However, during the expulsion of the contaminant, globules could be seen breaking the surface and then very rapidly dissipating in a manner indicative of the effect of surfactants breaking up oily compounds. Again, this was an excellent example of coal tar emulsification. A video addendum has been included in this report as a CD to provide a graphic example of the points that have been discussed regarding emulsification activities in the **MW-15** holder and injection area as well as the events that unfolded during the treatment of the **MW-13** pilot demonstration area.

The objective of the demonstration project in the **MW-13** area was to assess the potential of the technology to increase the efficiency of the extraction wells. Therefore, as was the case with the work in the **MW-15** area, comments on this aspect of the work could not be presented until sufficient time had elapsed for the data to be gathered and a rationale developed.

At the time of the April sampling event, it was reported that the production of coal tar from the extraction wells had been increased by six (6) fold over the average historical production prior to treatment. Although this was very welcome news, it did not tell the

whole story based upon observations made during the injection work. Of primary importance, is the fact that no provision was made to measure the volume of soluble or emulsified contaminants that were produced coincidental with the measurable free product globules. Therefore, a significant volume of ECT was no doubt extracted but, passed through the system undetected. It is paramount that a field testing methodology be developed or adopted to close this loop. It was graphically pointed out that groundwater samples collected and transported to an analytical laboratory unfortunately, do not “keep” in transit. Thus, they are not representative of the aliquot at the time of collection. Therefore, field testing is necessary to develop data representative of actual real time conditions.

Overall, application work in the **MW-13** area provided several challenges. Freezing weather required the protection of all machines

and hoses to avoid damage from the cold. Debris in the Filled Ravine and the unexpected high concentration of cobbles in the Copper Falls Aquifer, were responsible for an extraordinary number of refusals. Though expulsion of oxidation reagents were greater than expected, the TEAM did a very good job of containing the problems and preventing off-site releases to the environment.



Photo 36 reveals the communication between **IP-2** (near **E-1**) and extraction well **E-3** across the site. The force of the reaction gasses raised the water level in **E-3** from the Copper Falls Aquifer to above the well casing into the vault. An industrial vacuum is used to aspirate the groundwater and contain it in a tank. The bubbling from the well casing can be seen in the top center of the photo (see orange arrow). Coal tar can be seen floating on the surface (see yellow arrow). At this time, the pH of the groundwater was still at ~6. However, with in one hour of the expulsion, the pH had risen to ~7. Communication from **E-1** across the site to **E-3** demonstrated that conditions in the Copper Falls Aquifer were conducive to contaminant extraction.

MW-13 Summary:

The objective of the pilot demonstration located in the **MW-13** monitoring well nest was to determine if the *Cool-Ox*TM Process could improve the efficiency of the current pump and treat system to extract coal tar artifacts from the Copper Falls Aquifer.

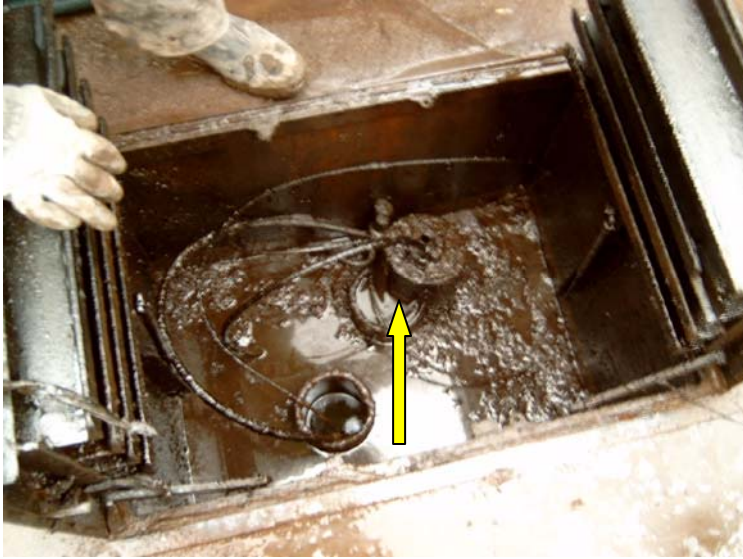


Photo 37 reveals that the color of the groundwater in extraction well **E-3** is changing from a semi-clear appearance to a dark tan, translucent liquid interspersed with globules of coal tar. Note the groundwater has been raised to the top of the casing (see arrow) and the pH is now approximately 7.5.

To implement this demonstration program, the Quality Assurance Project Plan (QAPP) and Scope of Work (SOW) called for the placement of twenty-seven (27) injection points (IPs) on a nine and one-half (9½) foot matrix within a forty (40) by sixth (60) foot (2,400 square feet), pilot demonstration area. The designed included, injecting 33,633 gallons of *Cool-Ox*TM reagent over a vertical injection interval from eighty (80) to thirty (30) feet below ground surface (fbgs), then grouting the IP to the surface with Portland cement. A total of 34,450 gallons was injected.

Because of interferences from utilities, building placements and the decision not to breach St. Claire Street with IPs, the injection matrix was abandoned in favor of strategically placing the IPs where they would, in the opinion of the TEAM, accomplish the stated objective. Because of the unanticipated concentration of cobbles in the Copper Falls Aquifer, only one IP (**IP-1**) reached the design depth of eighty (80) feet. The average depth, accounting for refusals, was sixty-three and one-half (63½) feet; approximately ten (10) feet above the bottom of the contaminant mass (see **Figure 2**). In spite of this problem, the TEAM was able to treat approximately seventy-eight percent (78%) of the targeted volume of contaminated soil (groundwater).

In spite of several breakouts occurring during the site work, all reagents and accompanying contaminants were contained with no off-site releases to the environment. However, there was evidence that the presumed integrity of the Miller Creek Aquitard may be in question. Because of the location and pattern of the breakout near the north side of the maintenance building coupled with the information developed with the discovery of a holder in the **MW-15** pilot area, there is speculation that the large holder shown under the maintenance building on the Sanborn Map, may in

fact, be in place. The settling of the sidewalk and the building wall prior to the pilot demonstration, would support this supposition.

Communication between **IP-2**, in the area of extraction well **E-1**, and extraction well **E-3**, provided evidence that communication and penetration of reagent throughout the Copper Falls Aquifer was apparent. This was major finding, lending credibility to the premise that the Aquifer could be treated with the Technology. The observations that a significant mass of contaminants was expelled to the top of the casing in extraction well **E-3**, and the emulsification of the coal tar contaminants, suggests that successful treatment of the Aquifer employing the *Cool-Ox*TM Process is quite possible.

One significant factor, not investigated during the pilot demonstration, was the lack of an attempt to apply remediation scale vacuum to the extraction wells. Instead, the Technology was depended upon to transport contaminants to the extraction wells (employing the Technology's physiochemical phenomenon) and the in-place extraction system to extract the treated moiety. Because of the differences in the larger capacity of the injection system to deliver reagents and the limited capacity of the current pump and treat system to extract the reacted contaminants, a significant dynamic imbalance existed. Although not a component of the

demonstration, it is the conclusion of the TEAM that if a vacuum system were employed to pull contaminated groundwater from the Aquifer in balance with the injection rates, an efficient cost effective remedial system could be implemented. The TEAM has given considerable consideration to design parameters of such systems, both for the remediation of the deep Aquifer as well as mitigation of holders and the filled Ravine.

The TEAM was gratified to learn that, subsequent to treatment, the productivity of the extraction of coal tar, by the current system, had increased by a factor of six (6). While this is certainly significant and very welcome news, the quantitative determination

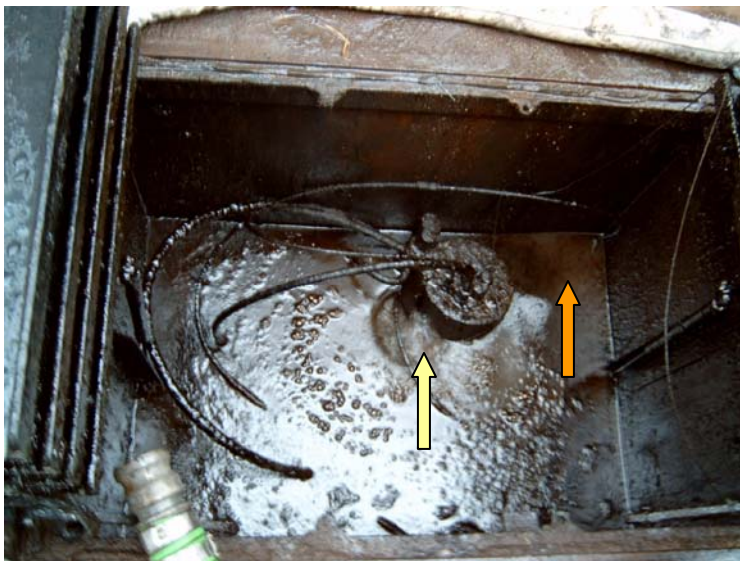


Photo 38 The pH of the groundwater in well vault **E-3** has reached 8, the optimum for the oxidation reaction of the *Cool-Ox*TM process to proceed. The color of the water is now a milky tan (see orange arrow) indicating the presence of emulsified coal tar (ECT). Note the coal tar has broken up to finer globules and foam (see yellow arrow) is now being generated from inside the well casing. The amount of ECT expelled from the well is increasing.



As the groundwater level in **E-3** casing drops, **Photo 39** reveals the ECT left in the vault. Note the foam (see arrow) generated as the oxidation reaction proceeds.

PAHs under similar conditions at other sites, the reduction can be quite significant. This factor would also contribute to the remediation of the Aquifer without the oxidized faction being extracted.

During the application process at the **MW-13** area, the question was asked by Mr. Winslow if “the TEAM believes that the site was in better shape after the injections than before?” The TEAM believes that the site is in better shape and that the knowledge gained will allow remediation to go forward with far greater cost savings than predicted.

of the mass of soluble or emulsified contaminant extracted, although believed to be considerable, is unknown and graphically points up the need to develop and employ field analytical methods of accountability. Similarly, since no baseline samples were collected to ascertain the chemical species distribution or concentrations in the groundwater, the effect of the application on the hydroxylation of target pollutants in that media is undetermined. However, if the effect at this site is consistent with the destruction of BTEX and

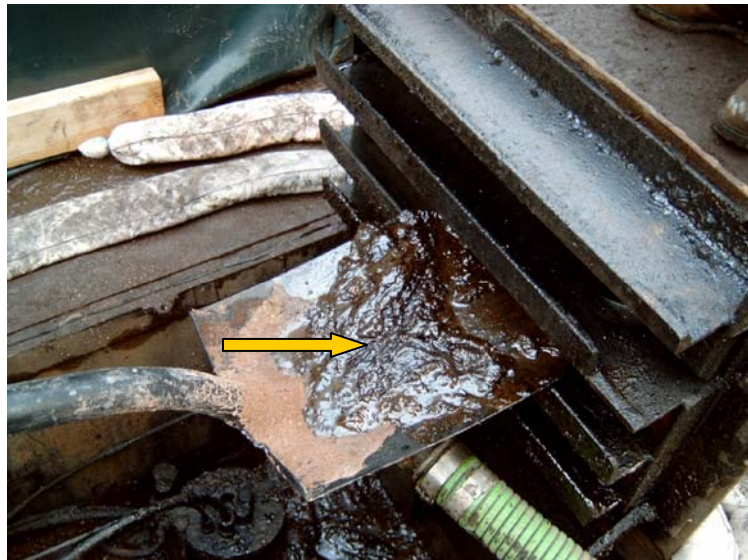


Photo 40 reveals the ECT grease-like material (see arrow) that was expelled from **E-3** by the reaction occurring in the Copper Falls Aquifer. As the globules of ETC were ejected into the vault, they settled to the bottom and congealed into the gelatinous material shown on the shovel. The material was containerized for disposal.



Photo 41 above shows the collector (see arrow) in the water treatment plant that is employed to separate the coal tar from the groundwater pumped here from the four extraction wells installed in the Copper Falls Aquifer. Although significant reactions were occurring in extraction well **E-3**, the expected changes in the appearance and chemical characteristics of the water in the collector, either in color or the pH, did not occur. Upon investigation, it was determined that the extraction pump in **E-3** was faulty. This discovery was critical to the project since fluctuations in the amount of coal tar captured in the collector was one of the parameters designed to measure the success or failure of the pilot demonstration.

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Figure 2

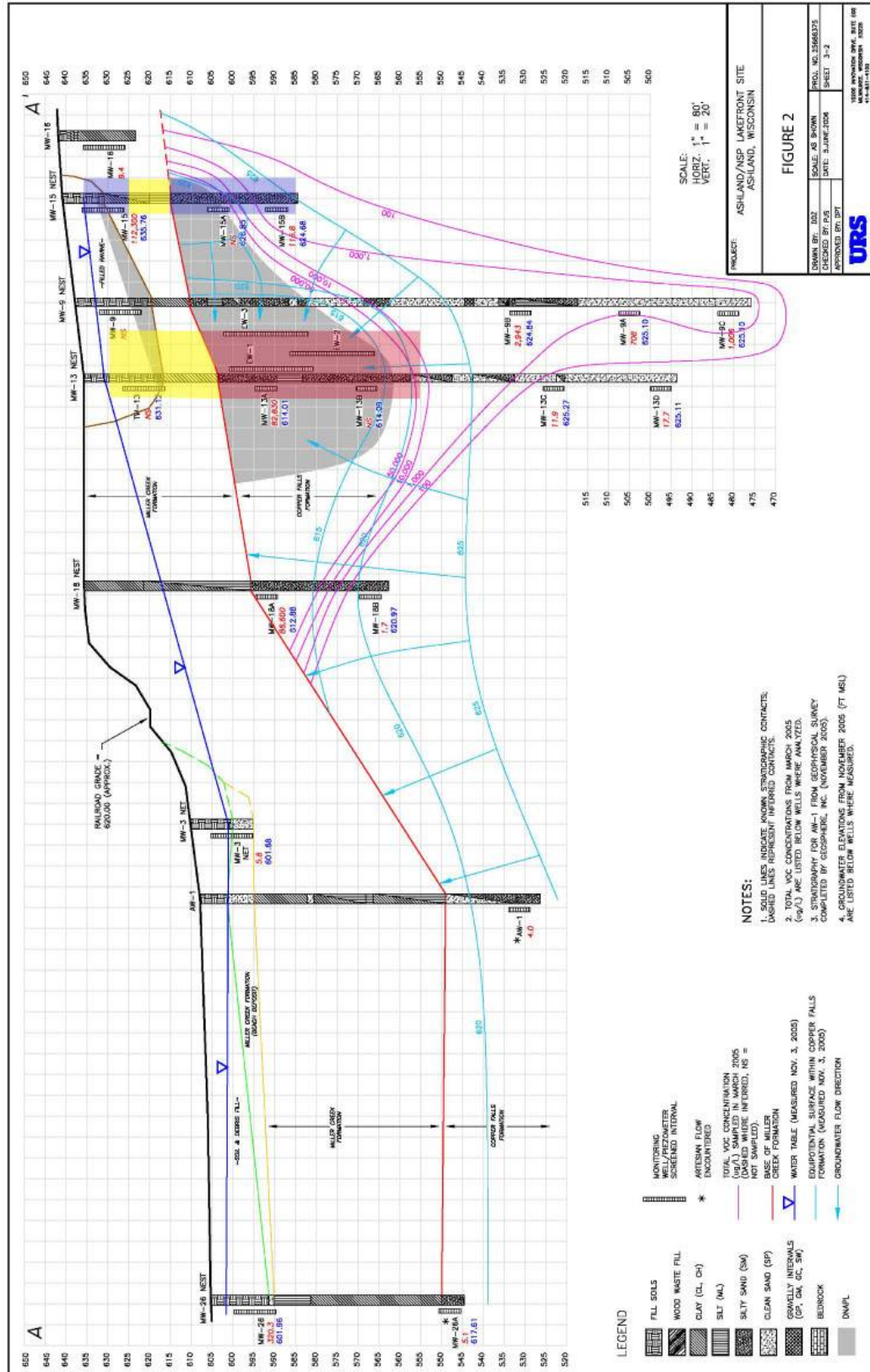


Figure 3

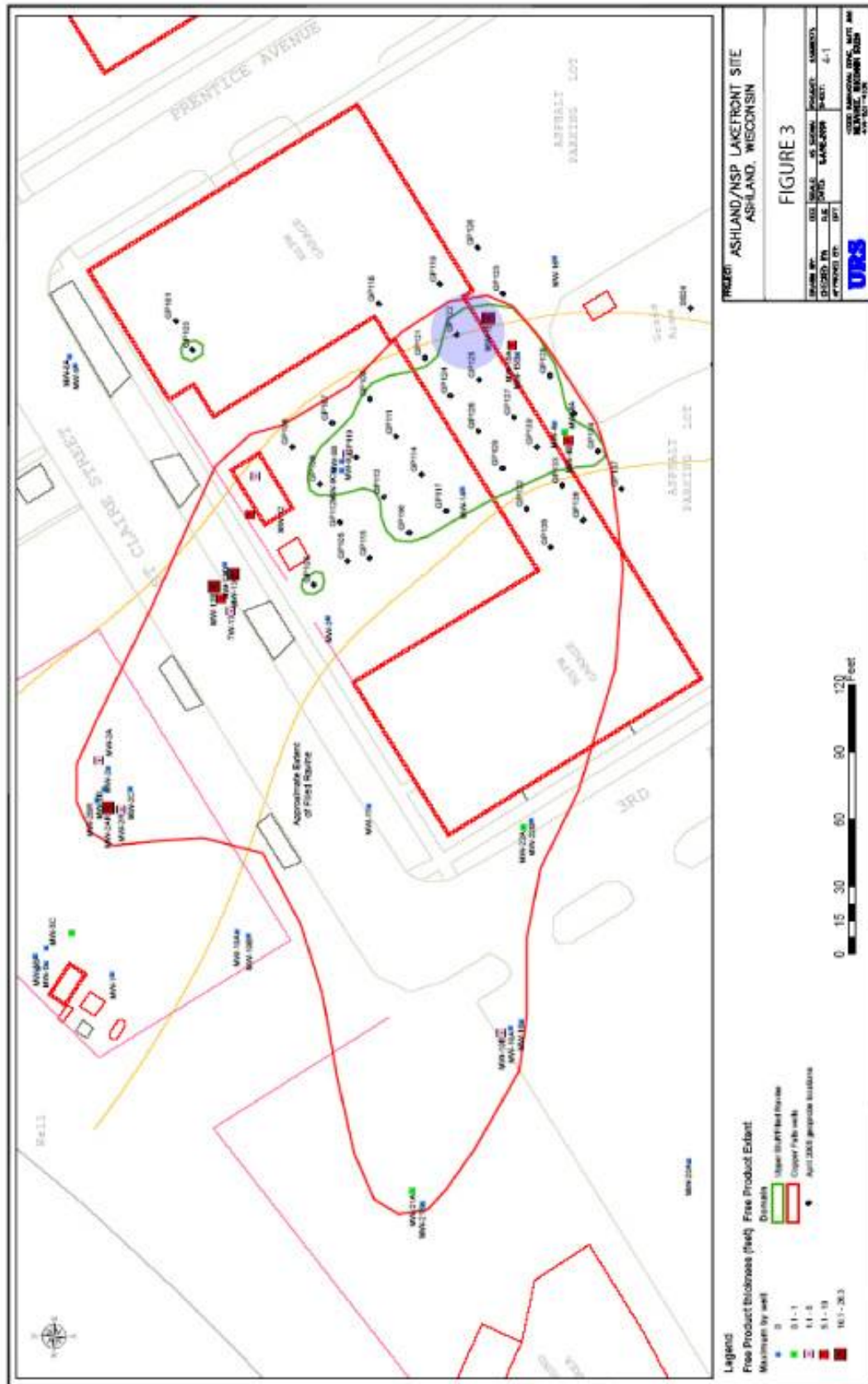
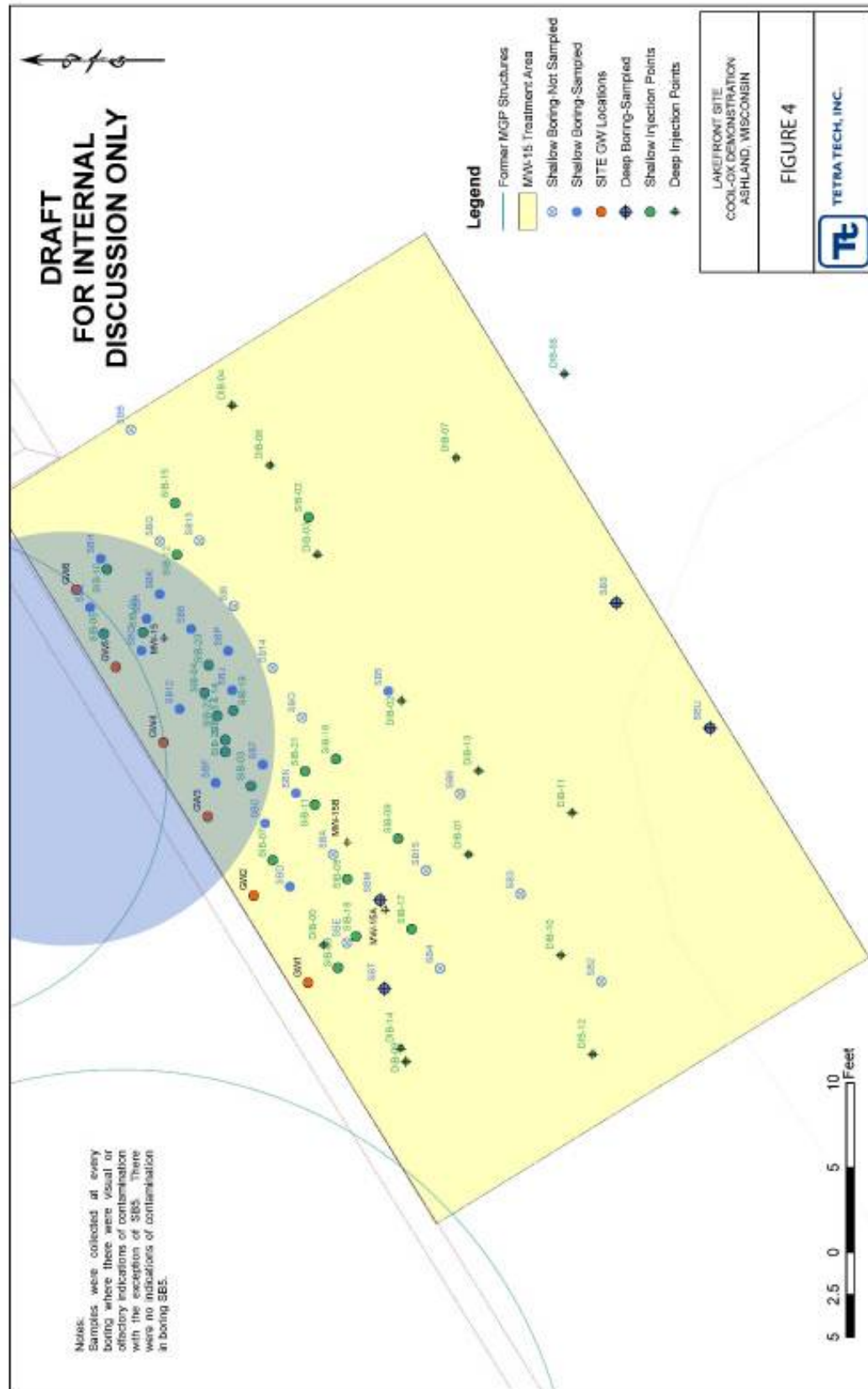


Figure 4



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Monitoring Well MW-15
December 1, 2006 - January 11, 2007

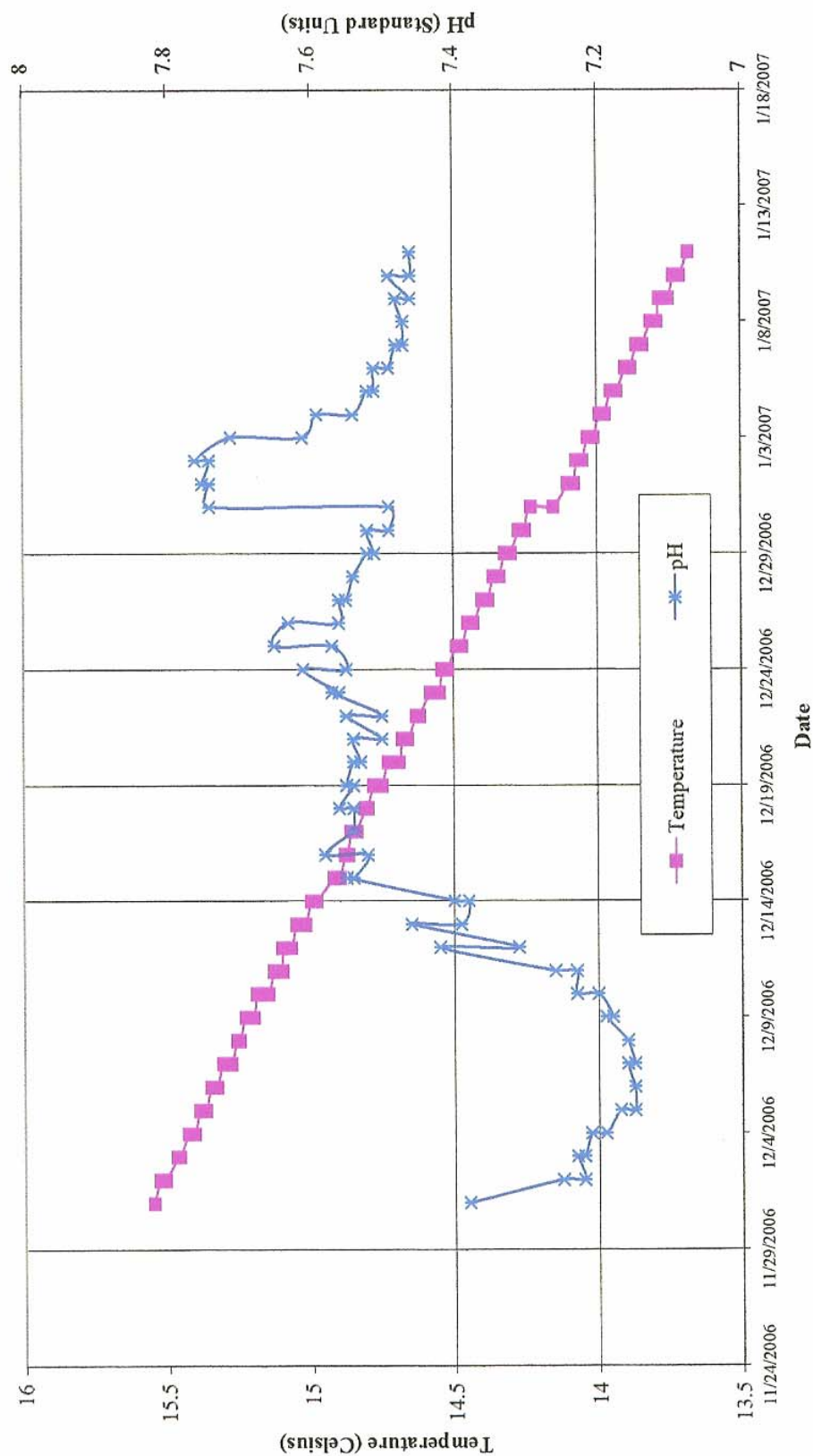


Figure 7

CHART 1: NSPW SITE MW15 GROUNDWATER - 24 HOUR HETEROTROPHIC PLATE COUNT: BEFORE AND AFTER COOL-OX TREATMENT

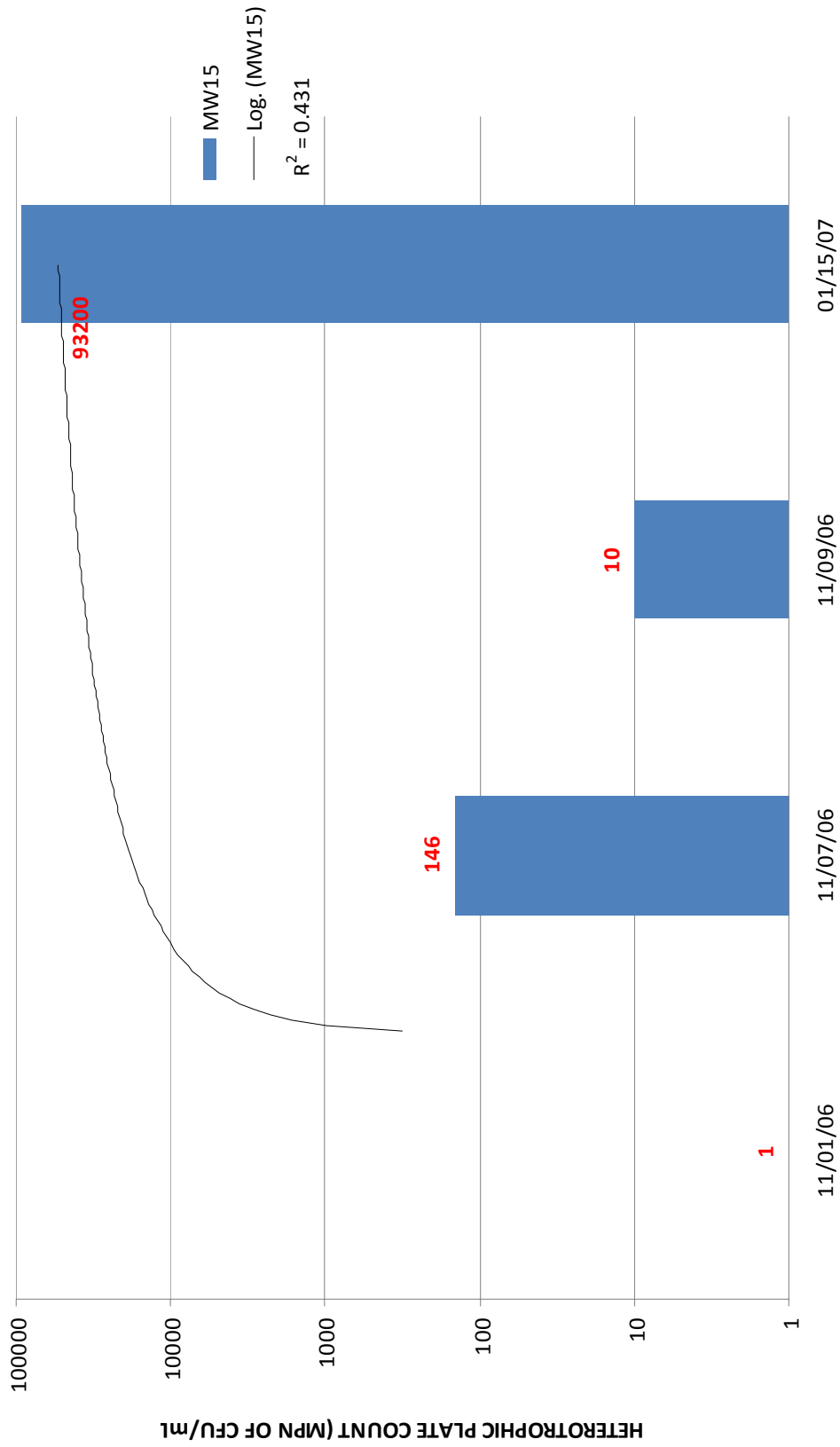


Chart 1

Chart 2

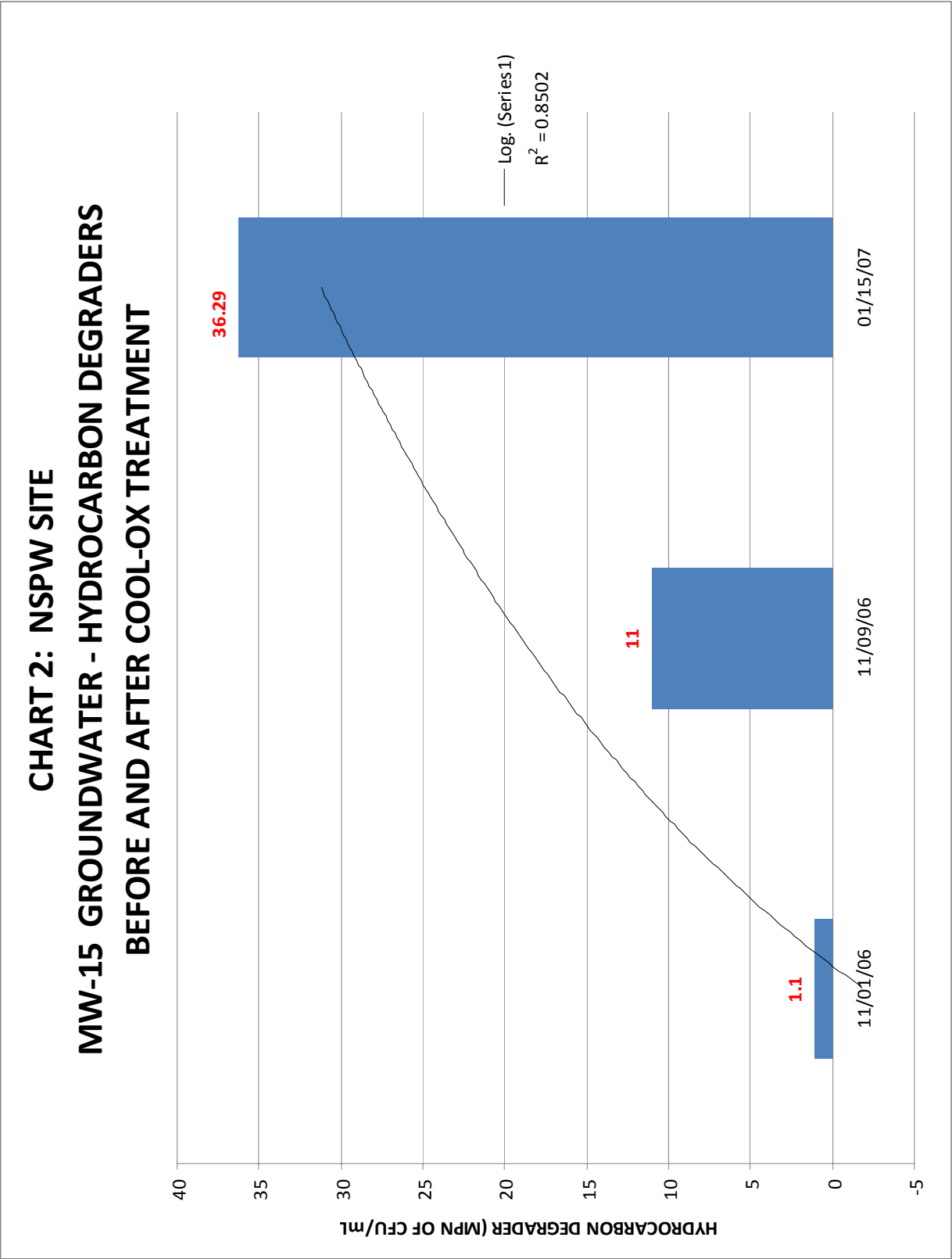


CHART 3: NSPW SITE
MW-15: GROUNDWATER - TOTAL PLATE COUNT
BEFORE AND AFTER COOL-OX TREATMENT

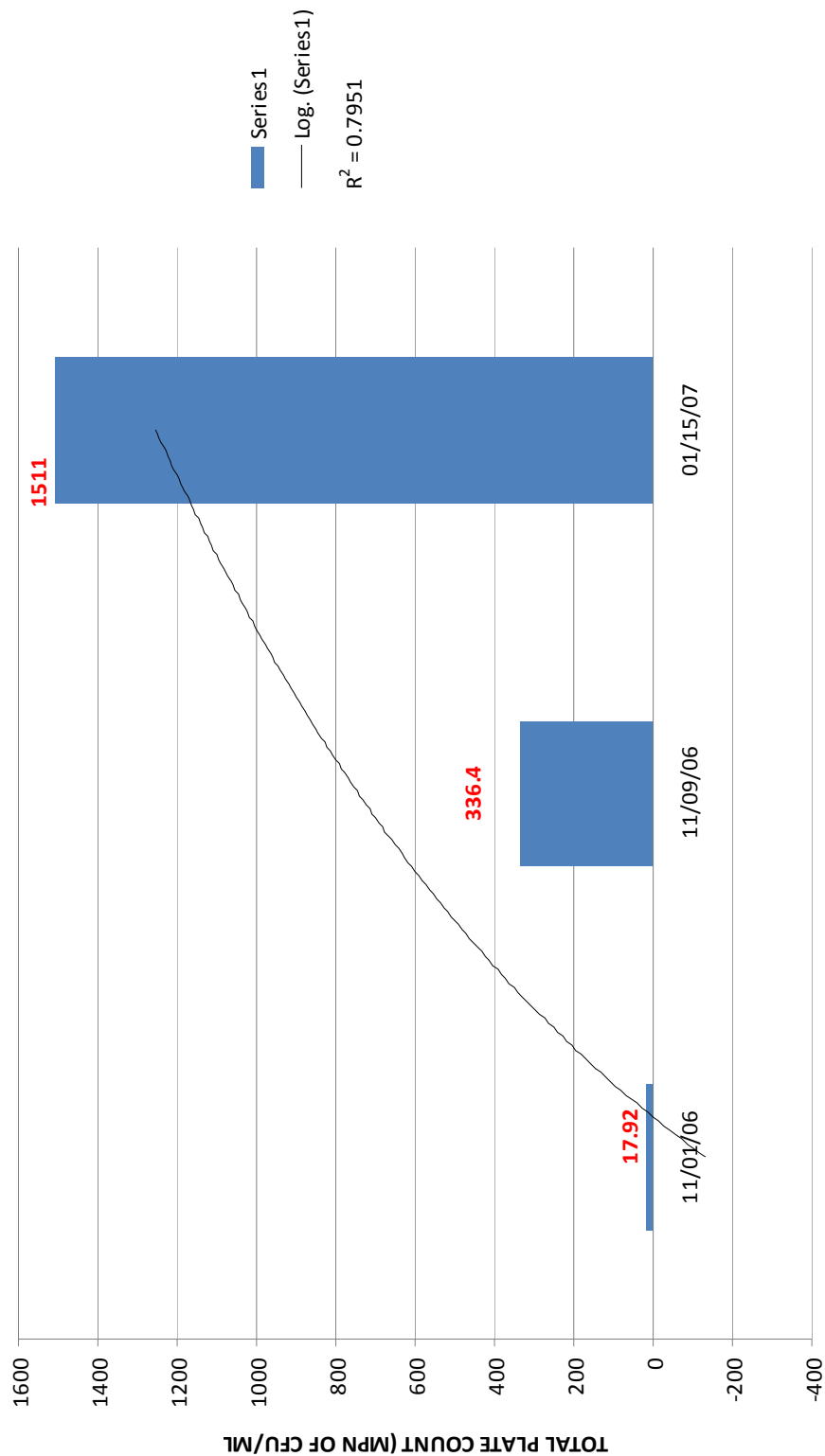


Chart 3

Chart 4

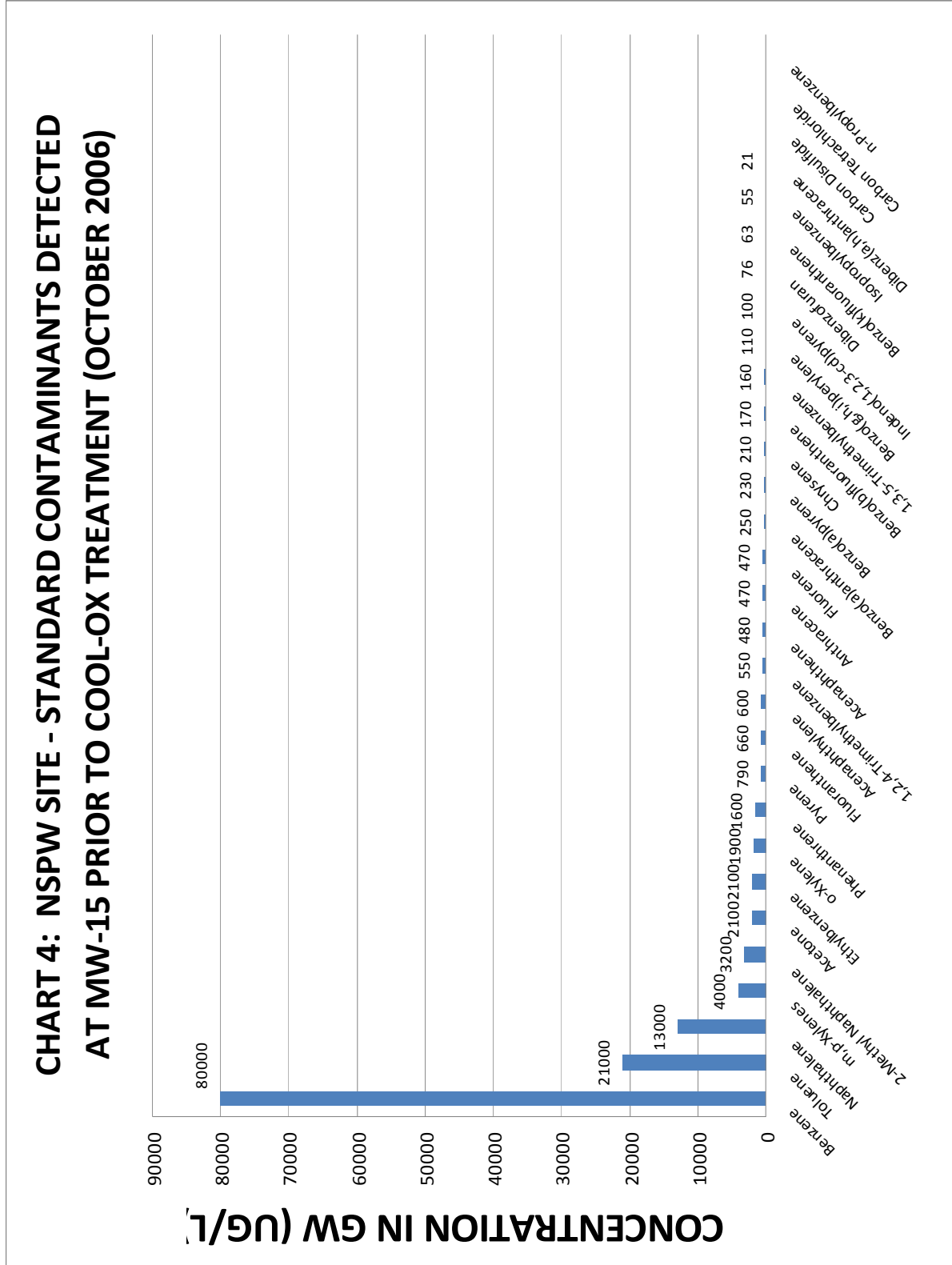


Chart 5

**CHART 5: NSPW SITE - STANDARD CONTAMINANTS
DETECTED IN MW-15 GROUNDWATER
FOLLOWING COOL-OX TREATMENT IN (JANUARY 2007)**

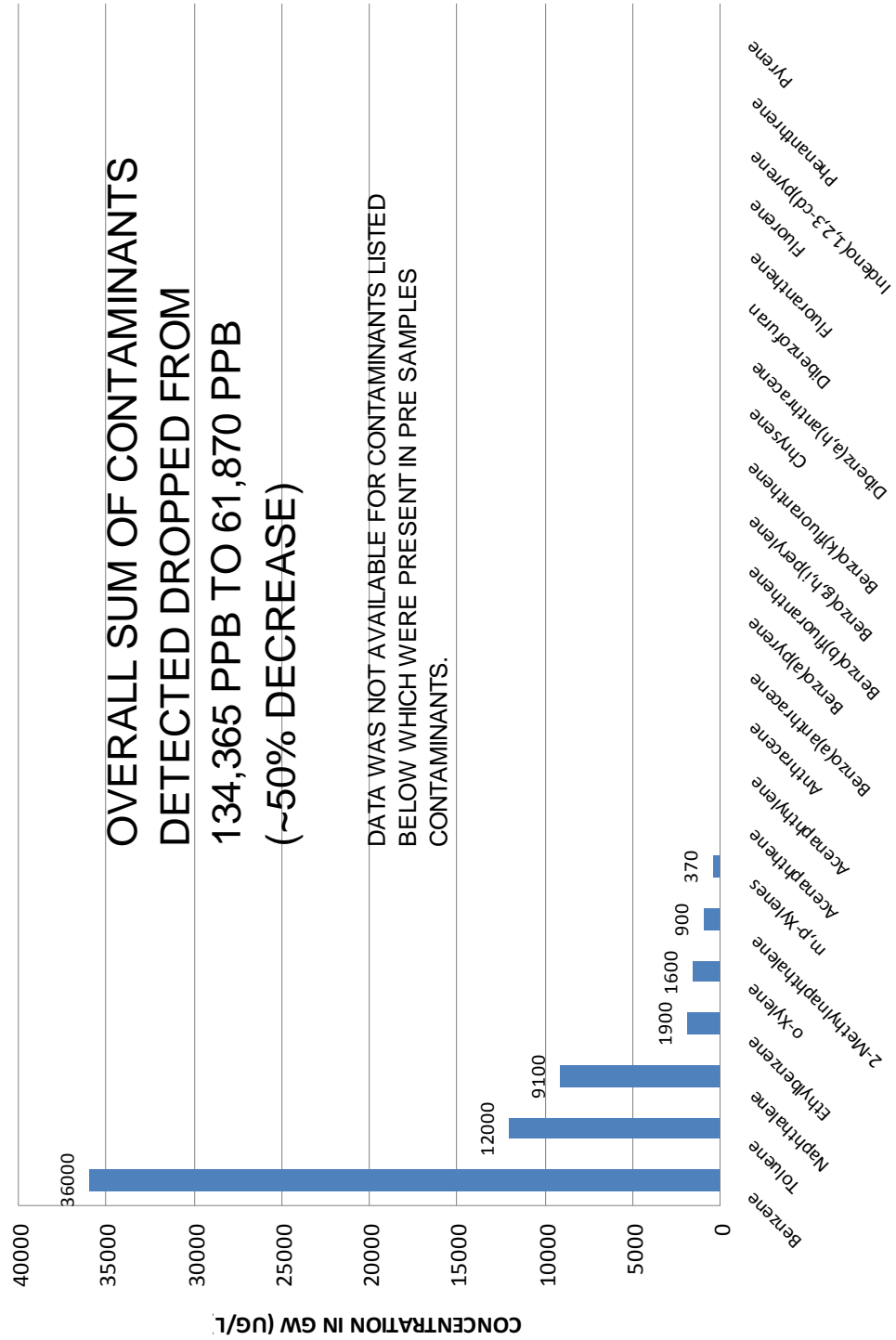


Chart 6

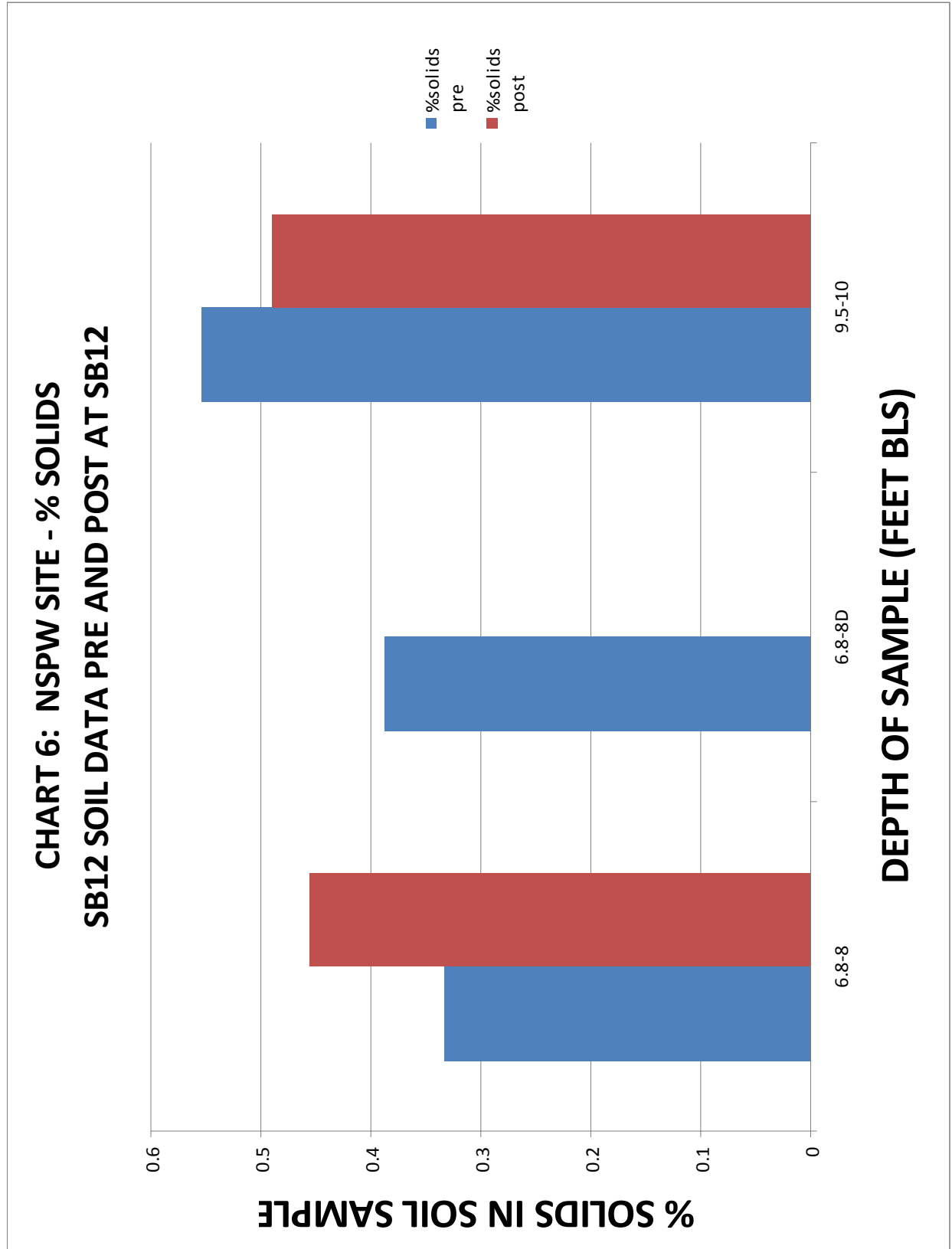
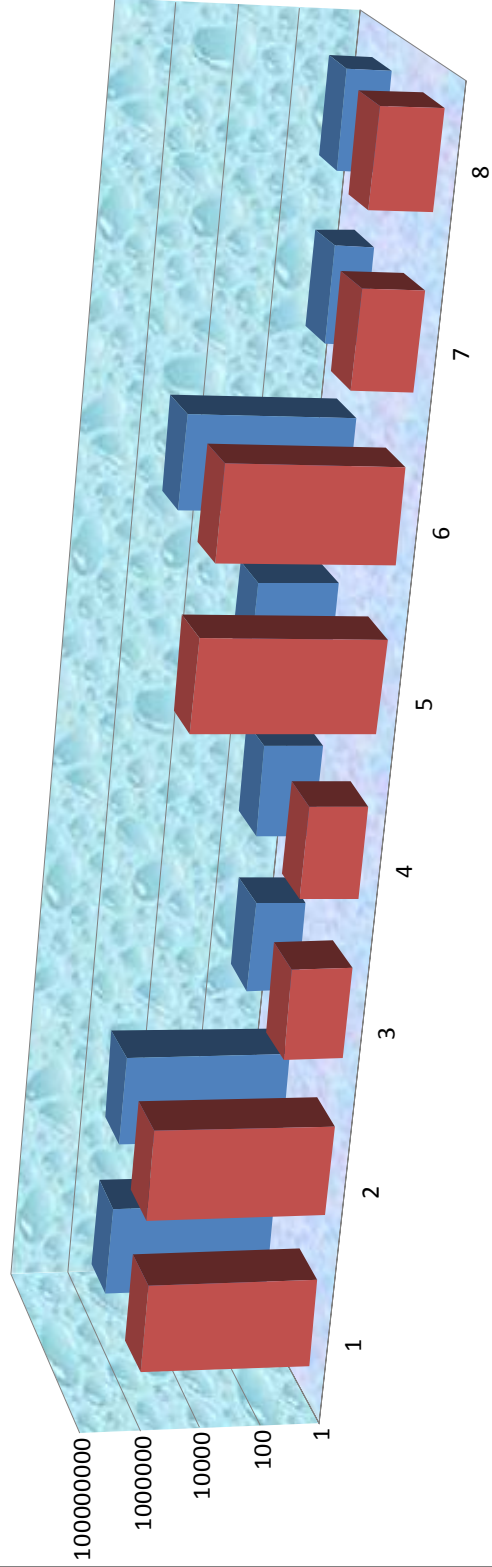


CHART 7: NSPW SITE
SB12 GASHOLDER TREATMENT AREA
SOIL-BACTERIA DATA BASELINE AND POST COOL-OX TREATMENT



POST COOL-OX	510000	1010000	89.59	89.59	1000000	505000	94.22	104.2
BASELINE	428000	428000	51.22	89.59	520	337000	19.65	26.35

THPC 24 hr: TOTAL HETEROTROPHIC PLATE COUNT (CFU/ml)

HD: HDROCARBON DEGRADERS (CFU/ml)

TPC: TOTAL PLATE COUNT (MPN of CFU/ml)

■ POST COOL-OX ■ BASELINE

Chart 7

CHART 8: NSPW SITE SBN OUTSIDE GAS HOLDER AREA BACTERIA DATA BASELINE AND POST COOL-OX TREATMENT

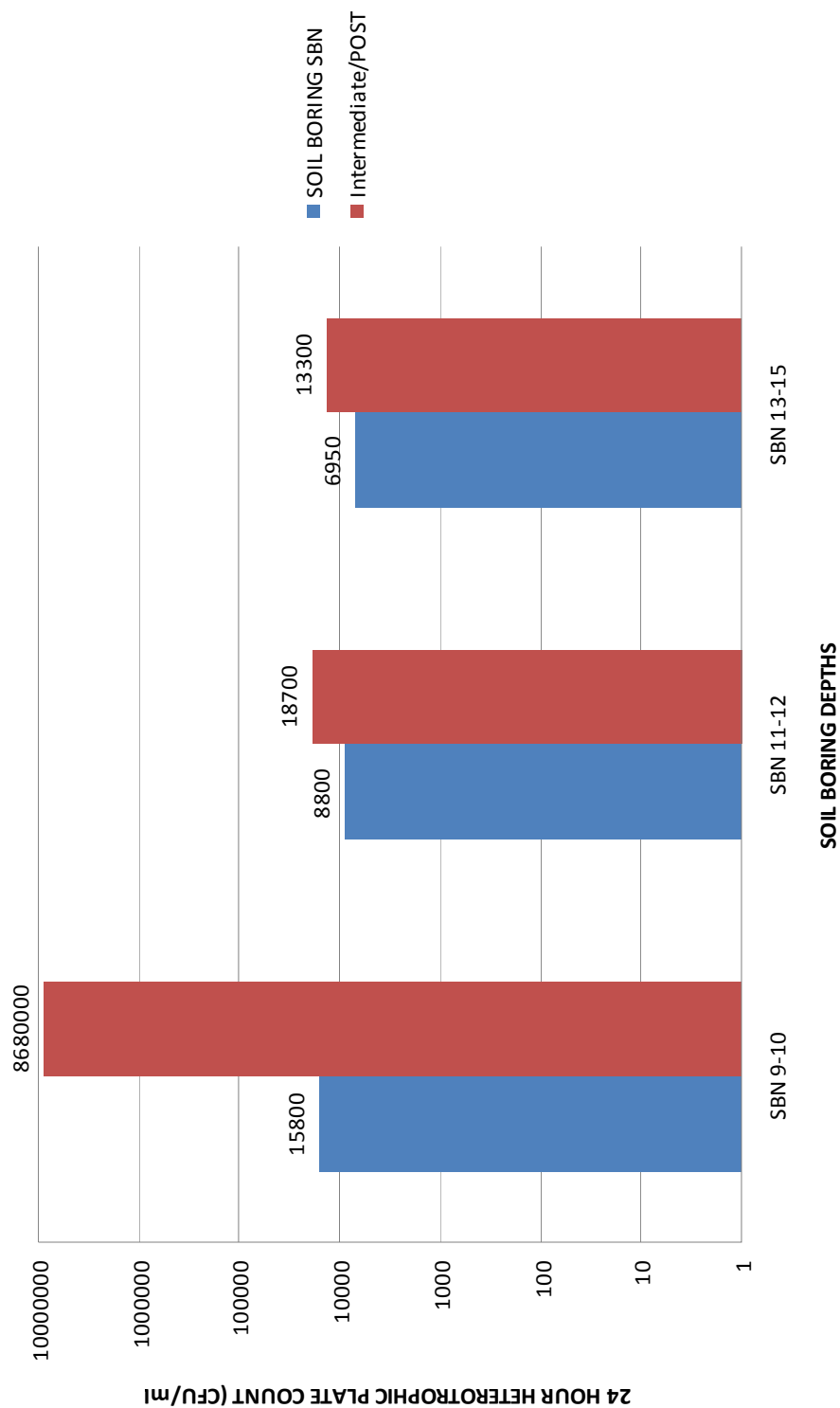


Chart 8

Chart 9

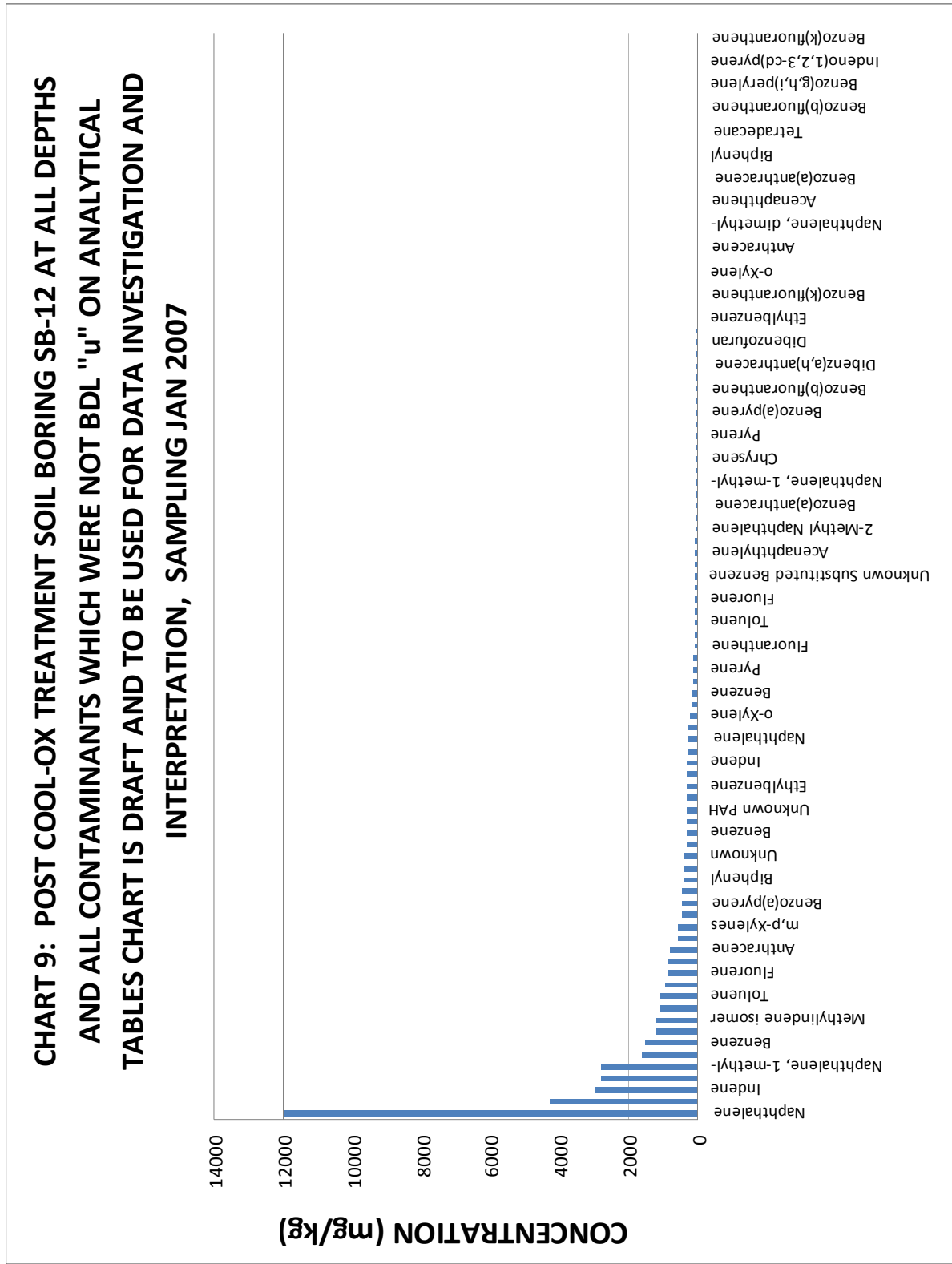


Chart 10

CHART 10: BASELINE SOIL BORING SB12 AT ALL DEPTHS AND ALL
CONTAMINANTS LISTED WHICH WERE NOT BDL "u" IN ANALYTICAL
TABLES
CHART IS DRAFT AND TO BE USED FOR DATA INVESTIGATION AND
INTERPRETATION, TDD/DTI

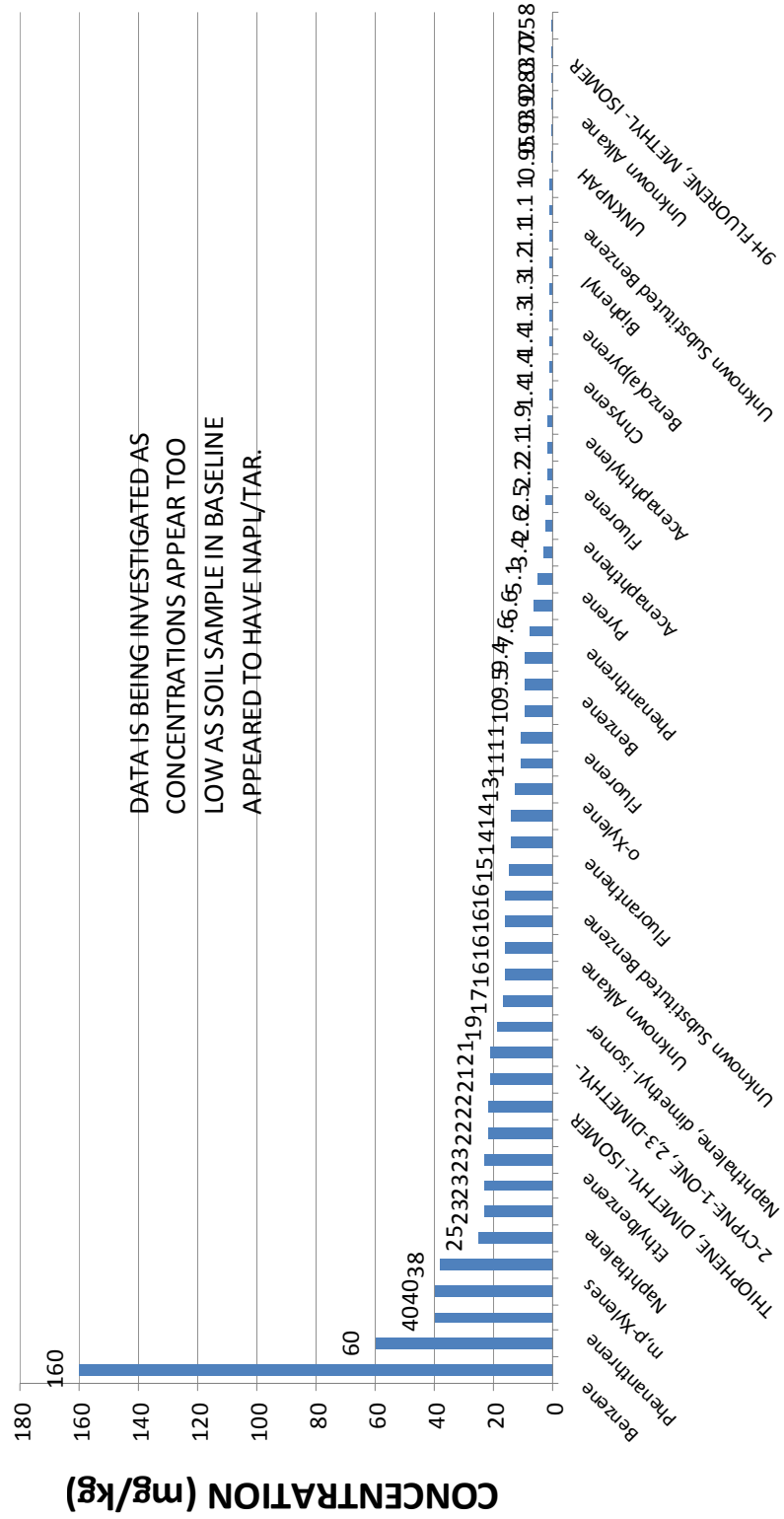


Chart 11

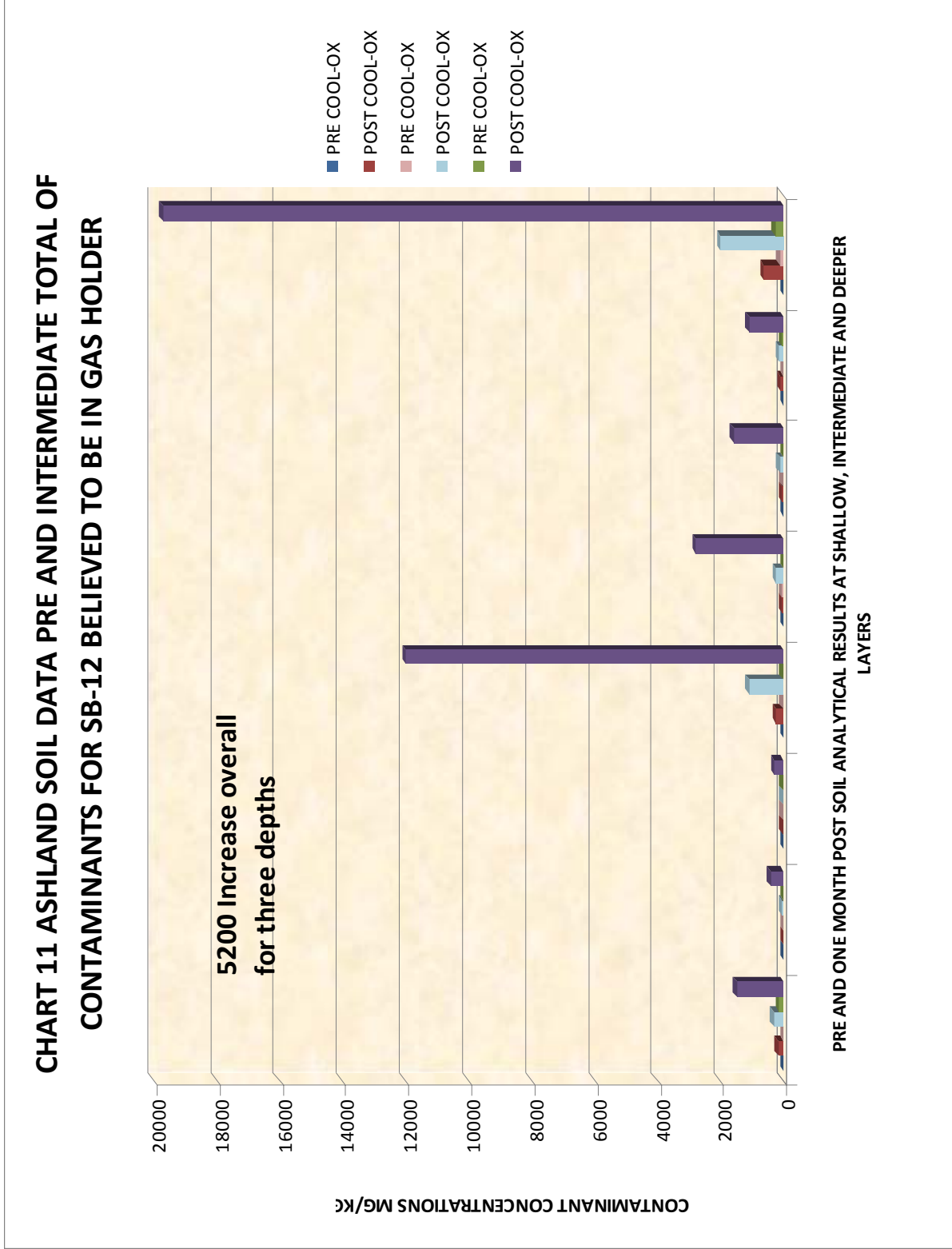


Chart 12

**CHART 12 ASHLAND SOIL DATA PRE AND INTERMEDIATE TOTAL OF
BENZENE TO TOLUENE CONTAMINANTS FOR SBN BELIEVED TO NOT BE IN
GAS HOLDER**

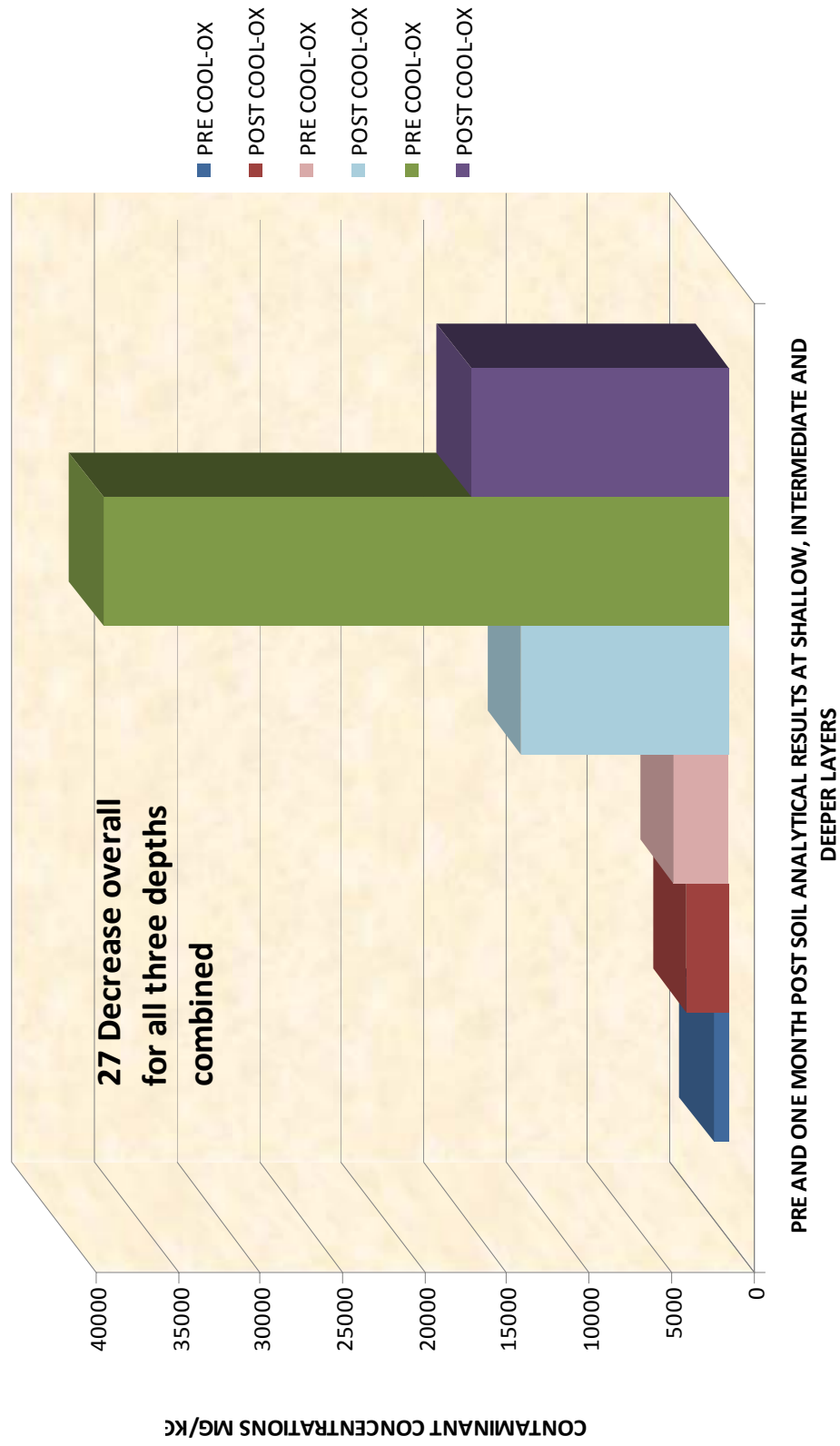


Table 1

Table 1 MW-15 Pilot Demonstration Area Shallow Application in Miller Creek Formation - Four (2) to Eighteen (18) fbgs					
Date	IP (SIB) ¹	Depth ²	Holder ^{3, 4}		Observations
			Inside	Outside	
14-Nov	1	18'	300		Odor - No Foam (ONF)
	2	18'		200	Influenced MW-16 & Sampling Point SBS (NONF)
	3	18'	300		ONF
	4	18'	300		ONF
	5R	8'			Refusal on Holder Wall
	5	18	300		ONF
	6	18		300	Odor and Foam (OF)
	7R	8			Refusal on Holder Wall
	7	18		300	Odor - Heavy Foam
	8	18		300	Odor - Heavy Foam - Product
15-Nov	9	18		300	Odor and Foam - Mild
	10R	6.5			Refusal - Holder Wall or Debris
	10	18	300		ONF
	11	18		300	Odor and Foam - Mild
	12	18	300		Odor - No Foam (ONF)
	13	18	300		Odor - Heavy Foam - Product
	14	18	300		Coal Tar Residue Expelled
	15	18		300	No Reaction
	16	18		300	Odor and Foam - Mild
	17	18		200	Influenced MW-16 & Sampling Point SBS (NONF)
16-Nov	18	18		300	Odor - Heavy Foam
	19	18	300		Coal Tar Residue Expelled
	20	18	300		Coal Tar Residue Expelled from SIB-19
	21R	10			Refusal - Probably Debris
	21	18	300		No Reaction very Mild Odor
	22	18	600		No Reaction very Mild Odor
	23	18	200		No Reaction very Mild Odor
	24	18	300		Coal Tar Residue Expelled from SIB-19
	25	18	300		Coal Tar Residue Expelled from SIB-19
			4700	2800	7500 Total gal MW-15 Area

Notes:

¹ Injection Point - Shallow Injection Boring (SIB)

² Maximum Depth - Feet Below Ground Surface (fbgs)

³ Gallons of Reagent Injected per IP

⁴ Injection Point Location Relative to Holder

Table 2

Table 2
MW-15 Pilot Demonstration Area
Deep Application in Copper Falls Formation - Twenty-five (25) to Forty (40) fbgs

17-Nov Collected Samples from MW-15A & B - resumed work after Thanksgiving Holiday

Date	IP (DIB) ¹	Depth ²	Gal ³	Observations
14-Nov	1	40	200	⁴ Heavy Sheen - Odor - Foaming, lost bottom 10' of rods
	2	36R	250	Refusal - Heavy Sheen - Foaming - Odor- MW-15B bentonite seal pushed up
15-Nov	3	40	250	Contaminant observed outside MW-15B casing - strong reaction
27-Nov	4	40	200	Slight foaming - no odor
28-Nov	5	40	250	Foaming - Heavy Sheen - More reaction at MW-15B
	6	40	200	Discontinued 10' spacing - Sheen - Mild reaction - communicate with DIB-4
	7	32R	200	Start south to Delineate GW plume - very slight reaction - slight odor
	8	36R	200	Slight reaction with DIB-7 - no reaction - concentrations very low - bio only
29-Nov	9	40	300	Heavy "Cow Pie" no reaction @ MW-15B - Breakout by bldg. foundation
	9-A	36	190	190 in DIB-9 - demo for State DNR visitors
	9-B	36	200	Additional Demo for State DNR visitors - Cow Pie after they left
	10	37R	200	"Cow Pie" heavy foam - odor
30-Nov	11	35R	300	"Cow Pie" heavy foam - odor
	12	33R	300	No Reaction
	13	35R	300	Strong Reaction - heavy foam
	14	37R	300	"Cow Pie" heavy foam - odor
			3840	Total Gal. MW-15 Deep

Notes:

¹ Injection Point - Deep Injection Boring (DIB)

² Maximum Depth - Feet Below Ground Surface (fbgs)

³ Gallons of Reagent Injected per IP

⁴ Encountered difficulty below 25 feet - Lost 10 feet of tooling (35 to 45 feet)

Table 3

Table 3
MW-13 Pilot Demonstration Area
Deep Application in Copper Falls Formation - Thirty (30) to Eighty (80) fbs

Date	IP ¹	Depth ²	Gal ³	Observations ⁴
7-Dec	1	80	300	Drove to 80' (TD) - PAV plugged, pulled rods, lost bottom 15', DTW-20', pH 5.5-6
	1	65'R	900	broke out at phone service, betonited point, old probe hole (NR-E1)(BU), pH of foam 8
8-Dec	1	65'	200	broke out at phone service, betonited point, old probe hole (NR-E1)(BU)
	2	30'C	0	rods capped and left, no injection - continue Monday
11-Dec	2	70'R	600	break out at phone service again, abandoned boring grouted, discovered communication with E3 water rose to top of casing, communication in E-3 across site in CFA, great news E-3 pH 5.5 to 6
12-Dec	3	68R	2,200	E-3, DTW-18.5', pH6, water to top of casing, CT stringy, pH changing to 6-7, water turning from clear to translucent, CT breaking up into globules & appearing more soluble, no change in pH (5.5-6) or CT appearance (black/oily) in WTP
13-Dec	3		1,700	E3 water milky to amber, pH 8, CT globules breaking up on surface of E3 casing indicating emulsification, micells forming (?) from surfactant from CT & PAHs, odor down, pH in collector in WTP 5.5-6, no change in CT or appearance, water still clear, #3 grouted, E3 pump suspected inoperable, no way to measure success, Kellog/Lundy stop work pending testing of E3 pump
14-Nov				telecon Kellog/Lundy/Trainor/Winslow halt work pending pulling pump.
15-Nov				E3 pump replaced - mechanism gone, down until Monday
18-Nov	4	35'C		E3 pump OK by 3PM, drove to 35' & capped until Tue.
19-Nov	4	68'R	1,400	At 600 gal water to top of casing, pH 8, CT breaking up, after 1,300 gal water in collector in WTP turning cloudy, pH @7-8, CT now has wrinkled appearance, no doubt that WTP is now getting water from E3, Breakout outside of casing in E3, heavy CT material in vault, apparent cinders, wood blocks in vault, large void opened up in west side of vault, sand was used to fill opening, ~1.5 cy needed, IP grouted after 1,400 gal
20-Nov	5	45'R	450	pumped 450 gal, breakout into E3 vault, point grouted, pH 8 in WTP, water reddish translucent, CT rough
21-Nov	6	55'R	600	At 600 gal, breakout again in E3 vault, decided to grout and let site equilibrate until after holidays
3-Jan	7	78'R	400	Tooling stuck, pumped 400 gal @ 1,200 psi, rods are plugged, left in place until later date.
4-Jan	8	45'R	450	no effect on #7 rods, still stuck, grouted IP
5-Jan	9	73, R	100	pumped 100 gal @ 900 psi, rods stuck and plugged, left in place over weekend, no affect at wells
8-Jan	10	3'R	0	worked on freeing rods in IP#9, rewired Probe for greater lift
9-Jan	9	73'R	1,950	pumped 1,250, no communication with any wells, pumped 750 gal, breakout at St. Claire St., entire crew worked quickly to contain effluent, no release to storm sewers
10-Jan	11	64'R	1,400	mild activity (bubbling) in E2 & ~4' raise in GW in well, foaming outside of casing in E3
11-Jan	7	78'	2,500	injected 2,500 gal, slight reaction in E3 vault, foam, grouted IP and moved
12-Jan				worked stopped to check integrity of E3 well casing
15-Jan	12	67'R	1,700	rod clogged, pulled to free, lost bottom 20' of rod, reinserted, grouted
	13	45'R	650	pumped 650, grouted
16-Jan	14	70-R	1,350	slight activity in E2, bubbling, ~4' raise in GW, grouted
			18850	

Table 3 Continued

Table 3
MV-13 Pilot Demonstration Area
Deep Application in Copper Falls Formation - Thirty (30) to Eighty (80) fbg

Page 2

17-Jan	15	75R	1,200	no noticable reactions, grouted
18-Jan	16	60R	2,000	no noticable reactions, grouted
19-Jan	17	60R	1,600	no noticable reactions, grouted
22-Jan	18	58R	1,400	breakout by building foundation in are of large holder, team all worked to contain release, no effluent escaped off-site, parking lot was cleaned and swept, then washed down with clean water, all material was contained
23-Jan	19	67R	800	slight reaction in E1 vault, injection stopped, grouted
24-Jan	20	75R	1,400	angle probe under street, reaction in MV-13, ECT bubbling out, captured with drum vacuum, large volume of emulsified CT coming out of MV-13, globules would immediately break up at surface indicating action by surfactant, effluent was milky
25-Jan	21	63R	1,800	no noticable reactions, grouted
26-Jan	22	45R	200	rods plugged, grouted
29-Jan	23	67R	800	rods plugged, grouted
30-Jan	24	60R	800	angle probe under street, reaction in probe rod ECT foam expelled
31-Jan	24		2,000	
1-Feb	25	67R	1,600	angle probe under street, reaction in probe rod ECT foam expelled
			15,600	18850 34,450 Total gallons Injected MV-13 Area

Notes:

¹ Injection Point

² Maximum Depth - Feet Below Ground Surface (fbgs)

³ Gallons of Reagent Injected per IP

⁴ Observations

TD-